

NEW BRUNSWICK, N. J.

October 17, 1972

R. Schaffner, Ph.D.
Director, Office of Product Technology
Bureau of Foods
Food and Drug Administration
Department of Health, Education, and Welfare
200 "C" Street
Washington, D.C. 20204

Subject: SHOWER-TO-SHOWER Brand Body Powder

Dear Dr. Schaffner:

As a result of a finding by Professor S. Lewin that a SHOWER-TO-SHOWER Brand Body Powder sample contained 5% chrysotile asbestos, we have carried out extensive studies on the sample in question obtained from Prof. Lewin as well as representative samples from production of SHOWER-TO-SHOWER Body Powder.

As you requested in our meeting of September 21, 1972, we are providing our data as follows:

1. Italian Talc Mine:

Reports by:

- (a) Prof. F. D. Pooley (University College, Cardiff, U.K.)
- (b) Atomic Energy Research Establishment
(A.E.R.E., Harwell)
- (c) Mining Institute, Torino, Italy
- (d) Prof. S. Lewin (New York University)

2. Investigation of SHOWER-TO-SHOWER Body Powder:

Reports by:

- (a) Prof. M. J. Buerger, (M.I.T.)
- (b) Prof. G. E. Brown (Princeton University)
- (c) McCrone Associates
(Dr. I. Stewart)
(Dr. W. McCrone)
- (d) Colorado School of Mines Research Institute
- (e) Prof. F. D. Pooley (University College, Cardiff, U.K.)
- (f) Prof. S. S. Pollack (Carnegie-Mellon University)
- (g) Mr. J. Schelz (Johnson & Johnson)
- (h) Dr. J. M. Wehrung (Sperry-Rand)

DEFENDANT'S
EXHIBIT
D-8372

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A review of the data shows that:

1. Italian Fontana mine does not contain chrysotile asbestos deposits.
2. The Italian talc powder from the Fontana mine used in SHOWER-TO-SHOWER Body Powder has been shown to be free of chrysotile asbestos by both Prof. Pooley and Prof. Lewin.
3. Investigations by all available methods carried out by experts failed to establish any chrysotile asbestos in SHOWER-TO-SHOWER Body Powder. The investigations included transmission electron microscopy and electron diffraction, x-ray scan, step scan, Guinier camera, differential thermal analysis (DTA), petrographic techniques including optical staining techniques, and scanning electron microscopy.

A review of the data by the various experts clearly shows that Prof. Lewin's finding is wrong and has no basis in fact. We would like to point out that a release of such untrue information will create a great deal of unwarranted alarm among the public and will cause serious damage to our reputation and business.

In view of the nature of the enclosed material and the comments regarding Professor Lewin's work, we request that this material be held in strict confidence by the employees of the Food and Drug Administration interested in this subject, unless we consent to other use of it.

On behalf of myself and my colleagues, I wish to thank you for giving us the opportunity to fully review our data on this subject on September 21.

Very truly yours,
JOHNSON & JOHNSON

W. Nashed
W. Nashed, Ph.D.
Director of Science Information

WN/cw

Attach.

bcc: Dr. R. A. Fuller
Mr. D. R. Clare
Mr. H. Stolzer
Dr. A. Goudie
Dr. R. Rolle
Dr. G. Hildick-Smith

Mr. S. C. Smoyer
Dr. T. H. Shelley
Mr. W. Steinberg
Mr. R. C. Stites
Mr. E. G. Vimond

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Prof. F. D. Pooley

J&J-0130534

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DR. FRED POOLEY

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Education: B.Sc. University of Wales (Cardiff)
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M.Sc. University of Wales (Cardiff)
1963 Mineral Separation

Ph.D. University of Wales (Cardiff)
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From Dr. F.D. Pooley
Department of Mineral Exploitation

FDP/MM

28th September, 1972.

Dear Dr. Shelly,

The mineralogical X-ray and morphological examination of the Italian mine samples has been in progress for over a year. The samples were all collected under my supervision at the Fontana mine, which is the same mine from which the 'Shower to Shower' sample was obtained.

The report of the examination contains a brief outline of the results obtained. No chrysotile was found at the mine or in the samples taken, some tremolite was located, but was not asbestiform in character and has not been detected in 00000 talc imported into Great Britain for the past year, nor in shipments dating back to 1949. I hope that it is of some interest to you.

Yours sincerely,

F.D. Pooley.

J&J-0130536



DEPARTMENT OF
MINERAL EXPLOITATION

UNIVERSITY COLLEGE
CARDIFF

AN EXAMINATION OF ITALIAN MINE
SAMPLES AND RELEVANT POWDERS

This document represents the completion report of the Italian mine samples and other powders supplied by Johnson and Johnson, Cosham, Portsmouth, to the Department of Mineral Exploitation.

The persons involved in the examination of the material reported here were:

Mr. J. Lightfoot
Mr. G.A. Kingston
Dr. F.D. Pooley

Received: 8th September
1972

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REPORT OF INVESTIGATION OF ITALIAN MINE
SAMPLES AND RELATED POWDERS

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Introduction

Talc is hydrated magnesium silicate ($Mg_3Si_4O_{10}(OH)_2$) which can occur in a number of forms. In its compact form it is known as stealite or soapstone. The form normally employed for toilet purposes is soft and very friable in character. It is mined in many parts of the world including the U.S.A., Canada, France, Italy, Norway and India, as well as several other countries. It occurs in both a flaky and lath like form and the chief deposits occur in altered magnesia-rich calcareous rocks such as dolomite, marble, and magnesian limestone. The purest talc deposits occur in association with dolomite and marble. Talc also occurs in altered basic rocks such as serpentines and again as thin beds in mica schists. Commercial talcs contain a number of related mineral impurities. They may include antigorite (hydrated magnesium silicate) magnesite or members of the magnesite-chalybite series of carbonates, dolomite (calcium magnesium carbonate), tremolite and actinolite (calcium, iron magnesium silicates), chlorites (magnesium aluminium iron silicates) and other minor minerals such as the sulphides and spinels.

The hand specimens examined in this report were collected at the Italian mine and do not represent an average collection of specimens of material being produced at the mine. The specimens were collected with the intention of sampling those areas with obvious non talc mineral inclusions. Specimens were retained which showed differences in physical appearance, i.e. fibrous, flakey, massive and powdery in texture. Specimens of ore in which colour variation was observed were also collected. In general the colour of the talc ore varied from grey through white to a light green colour. Obvious inclusions in the talc ore itself were retained and a careful search at the various sample locations in the talc seam was performed for fibrous amphibole minerals.

Specimens of the hanging and footwall were also collected to assess their mineral content as these were likely sources of ore contamination, although the method of mining which consisted of hand filling methods precluded any gross contamination of the ore.

The hand specimens have been, where possible, prepared for examination by the optical microscope and both polished blocks and thin sections of material have been employed. Representative fractions of all hand specimens have been reduced to powder form and subjected to powder X-ray diffraction examination. The representative powdered samples also form the samples for morphological examination by the electron microscope.

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The list of samples obtained from the Italian mine are given in Tables 1 and 2 and throughout this report the samples are referred to by the preceding code number for each specimen.

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The objective of the examination has been mainly to establish the major minerals which occur in association with talc at the Italian mine. In particular to look at the association of these minerals with the talc and especially those minerals which are of the same family as the commercial asbestos minerals, i.e. the amphiboles and serpentines.

The objective of the optical examination has been to establish textural and mineral relationship and not to quantify the phases occurring in each hand specimen. X-ray work has been aimed at establishing the minerals observed by optical means and to produce reference patterns for future investigation together with computed data from pattern measurement.

Electron microscope work has been selective in nature and performed on the finer fraction of the powdered specimens. Its aim has been to describe the morphology of the particles produced by comminution of the hand specimens and to investigate any obvious structural information which might be of use in identification of individual mineral particles.

Representative data obtained from the various examinations are included in the following report.

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TABLE 1
LIST OF ITALIAN MINE SAMPLES

<u>Code No.</u>	<u>Description</u>
I.1.	Talc from footwall contact
I.2.	Sorting pieces (with obvious colour differences)
I.3.	Coloured talc (green)
I.4.	Face 10 sample with obvious amphibole inclusion.
I.5.	General ore
I.6.	Suspected Quartz sample
I.7.	Mica schist specimen
I.8.	Massive talc
I.9.	Grey talc 1st face
I.10.	Granular talc sample
I.11.	Carbonate and talc
I.12.	Footwall sample? Amphibolite
I.13.	Inclusion showing passage into talc bottom transit.
I.14.	Inclusion in talc seam face 4, middle of seam.
I.15.	Talc footwall contact
I.16.	Inclusion from face 1.
I.17.	Footwall rock sample
I.18.	Face 3 carbonate/talc sample
I.19.	Tremolite/quartz/talc sample
I.20.	Amphibole sample from Gianna level 1212
I.21.	Inclusion from face 2.
I.22.	Carbonate/talc sample
I.23.	Black gneiss 2 ft below talc seam
I.24.	Talc next to carbonate face 2.
I.25.	Footwall limestone
I.26.	Talc inclusions

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Table 1 Continued

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<u>Code No.</u>	<u>Description</u>
I.28.	Quartz/talc sample
I.29.	Sample 6 footwall
I.30.	Quartz/Carbonate/talc sample
I.31.	Black inclusion face 1
I.32.	Face 2 inclusion from base of talc
I.33.	Talc from lower left end of working
I.34.	Marble/tunnel wall
I.35.	Massive carbonate from rear end of working
I.36.	Grey talc specimen
I.37.	Carbonate in talc inclusion
I.38.	Pyrite/talc specimen
I.39.	5" - 0 pieces from crusher
I.40.	Platey talc
I.41.	Face 2, good specimen
I.42.	Face 1, coloured green (talc)
I.43.	Face 10, fibrous sample
I.44.	Face 1, pure talc?
I.45.	Face 1, good specimen
I.46.	Face 3, coloured specimen

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TABLE 2OTHER SPECIMENS EXAMINED

<u>Code No.</u>	<u>Description</u>
B1	Pure talc 1st face
B2	Greenish talc 1st face
B3	Talc 6 inches above footwall
B4	Talc from above inclusion
B5	Inclusion in talc
B6	Talc 2 ft above inclusion
B7	Section 2 ft above inclusion
B8	Pure talc 1st face
B9	Grey talc 1st face

Also examined

- 1) Batch shipments of ~~00000~~ talc
 - 2) Old samples of British powders.
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OPTICAL EXAMINATION OF SPECIMENS 11 - 146

Thin and polished sections were prepared of the specimens of wallrock and, where possible, the talc ore.

The minerals which formed a major constituent in at least one of the sections were quartz, muscovite, talc, chlorite, (var sheridanite), calcite, garnet, and tremolite; the latter only occurred as a major constituent in section 119. Phases which were always minor or accessory were microcline, plagioclase, biotite, pennine, epidote, clinozoisite, hornblende, actinolite (section 17), rutile, and opaque constituents pyrite, pyrrhotite, and chalcopyrite.

The identification of the minerals in the sections of these specimens was based on the optical characteristics of the minerals in transmitted and reflected light, both under plane polarised light (PPL) and crossed nicols (XN), combined with the results of the X-ray diffraction study of the crushed hand specimens. In some cases material was extracted from the sections and examined in R.I. liquids as in determining that the most common chlorite mineral in these specimens is a variety called sheridanite having a R.I. ω equivalent of 1.590 ± 0.005 and a birefringence of $0.012 - 0.014$. Similarly much of the muscovite was nearly uniaxial with a R.I. of 1.600 corresponding to the variety phengite, an abnormally siliceous muscovite. In the case of talc its confident determination optically is difficult since its optical properties are identical to muscovite. However, it was found that the common "feathery" form of the talc combined with the invariable occurrence of minute transparent inclusions (suspected to be silica) in the talc producing a 'dusty' appearance in thin section and a greenish colour in hand specimen, enabled talc to be distinguished from muscovite. Talc also exhibited slightly higher order interference colours in general. Where talc was only an accessory mineral to muscovite, as in some of the wallrock samples, then it could not be distinguished with certainty.

In the following pages (no. 7 to 48) the Italian specimens are systematically described as regards their mineral composition and mode of intergrowth. Numerous photomicrographs taken under PPL and XN are provided with the description to mainly illustrate the rock textures which, it is hoped, will provide information useful in the comminution of particularly the talc ore samples, and also displays the non occurrence of asbestiform amphiboles in the talc ore.

Specimen I1

Specimen I1 consisted of several pieces of wallrock with one piece displaying the talc/footwall contact. One polished section was made of the talc/footwall contact and one thin section of the wallrock alone.

The wallrock is a schist which in thin section displayed a segregation of the main minerals into thin lenticular bands composed, as in Figure 1, of long tabular aggregates of intermixed muscovite (var. phengite) and chlorite (var. sheridanite), and granular quartz exhibiting a polygonal grain boundary structure. Accessory rutile occurs as orientated inclusions in the chlorite and muscovite, and also opaque constituents which in polished section were identified as dominantly pyrite metacrysts with minor pyrrhotite. Some subhedral porphyroblasts of plagioclase also occur.

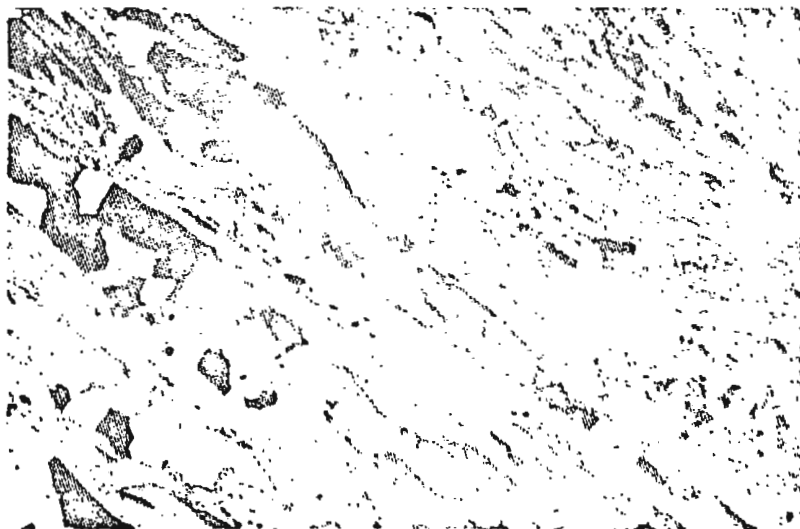


Fig. 1. Photomicrograph, X 40, of thin section of wallrock I1 under crossed nicols. A schist of quartz (granular white-black), muscovite (lamellar yellow-blue), and chlorite (lamellar white-blueish grey).

Specimen I3: 'coloured talc'

The minerals composing this specimen are major talc and chlorite (var. sheridanite) with the talc content much greater than chlorite, together with accessory garnet, rutile, and an unidentifiable finely dispersed phase occurring as minute transparent inclusions along the cleavage planes and grain boundaries of the talc and imparting a dusty brown appearance to the talc in thin section and a greenish colour in hand specimen. The talc occurs as medium grained feathery aggregates which are in places 'dusty' and grade into 'clean' transparent aggregates which are free of any inclusions. It appears that some retrograde metamorphic process has caused

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minor chlorite is dispersed in the talc matrix as small lenticular and globular fibrous aggregates. Rare garnet, possibly a member of the ugrandite series because of its anisotropy, occurs as subhedral porphyroblasts.

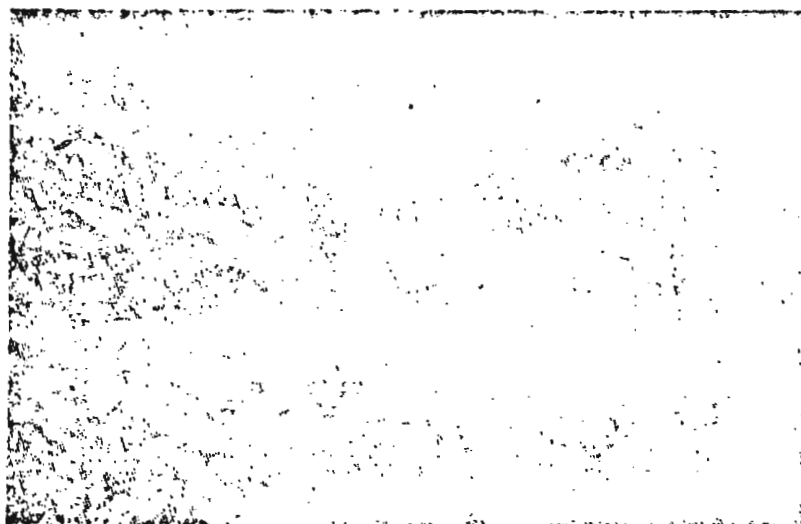


Fig. 2. Photomicrograph, X 24, of thin section of 'coloured talc' specimen I3 under crossed nicols. Dominantly talc (yellow-blue interference colours) showing murky brownish black patches due to presence of fine unidentifiable inclusions.

Specimen I5: general ore

A coarse aggregate of curving foliaceous and feathery crystals of talc displaying evidence of shearing and translation twinning. As in specimen I3, dusty inclusions of a transparent mineral with a general prismatic habit occurs dispersed in the talc. As before, but to a lesser extent, the talc is cleansed of these inclusions along zones associated with deformation and translation twinning, and it appears that the inclusions have either been converted to talc (as in the conversion of tremolite to talc by low temperature CO₂ metasomatism) or incorporated into the talc structure as a result of retrograde deformation metamorphism. Rare small subhedral garnet porphyroblasts also occur.

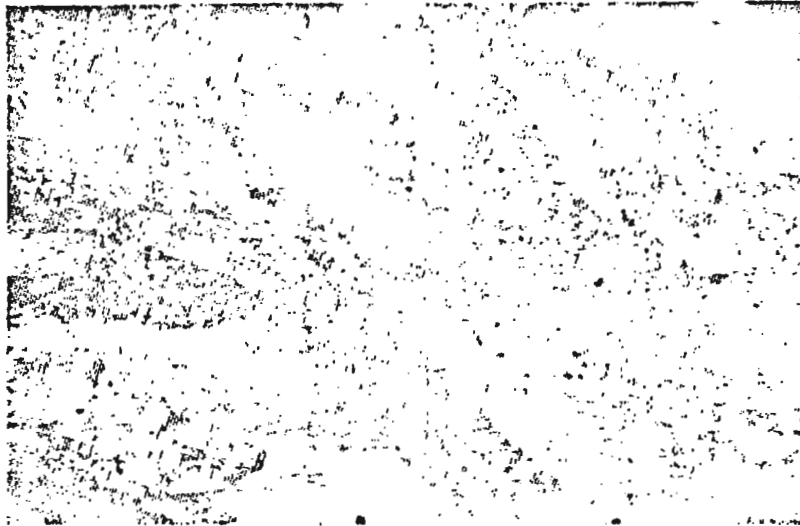


Fig. 3. Photomicrograph, x 24, of thin section of 'general ore' specimen I5 under crossed nicols showing the texture of the talc, and the 'murky' inclusion-rich talc compared to the clear inclusion-free talc.

Specimen I6

Specimen I6 consists of a very coarse aggregate of interlocking anhedral magnesite grains which exhibit strongly irregular and angular penetrating grain boundaries. The magnesite is characterised in thin section, Fig. 3a, by its marked change in relief and perfect rhombohedral cleavage in plane polarised light, and very high order interference colours, Fig. 3b, under crossed nicols.

Intergranular pockets of fine grained foliaceous and radiating prismatic crystals of talc together with rare chlorite (var. sheridanite) occur. In places the prismatic clusters of talc appear to have formed at the expense of the magnesite, perhaps as a result of a retrograde thermal metamorphism with its formation being ascribed to a reaction between the magnesite and silica. One subhedral porphyroblast of plagioclase feldspar occurs in the thin section.

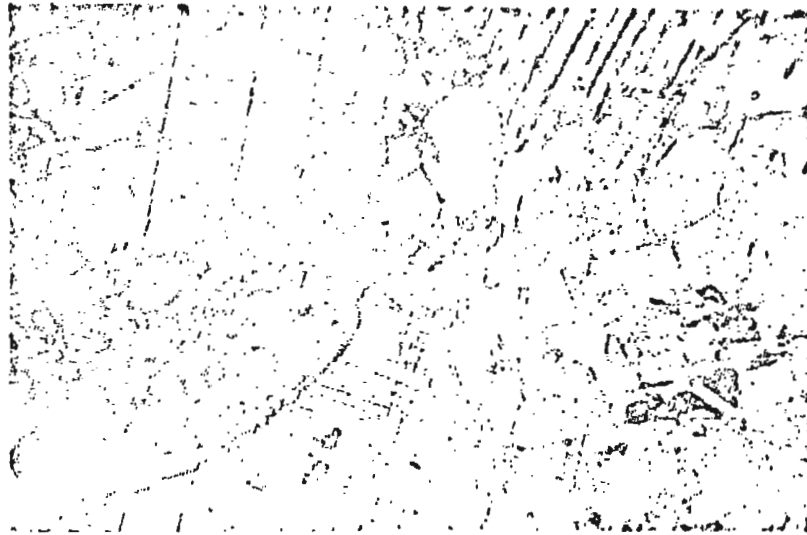


Fig. 3a. Photomicrograph, x 24, of thin section of specimen I6 under plane polarised light, consisting dominantly of magnesite with minor talc and rare chlorite.

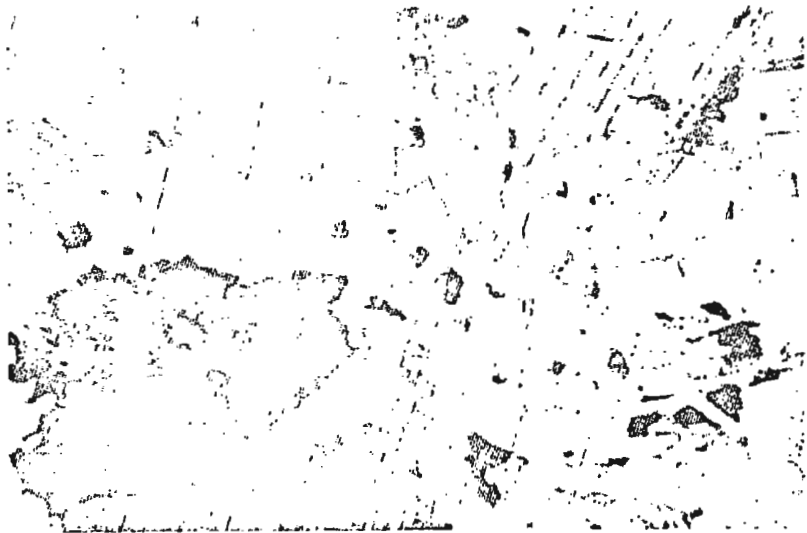


Fig. 3b. Photomicrograph of thin section of specimen I6, mag x 24, under crossed nicols showing the occurrence of small equigranular and prismatic crystals of talc penetrating and interstitial to coarse anhedral magnesite.

Specimen I7

This specimen of wallrock is a quartz-muscovite-garnet schist (Figs. 4a, 4b, and 4c) containing some accessory actinolite, brown hornblende, talc and rare biotite.

The muscovite (var. phengite) forms long lenticular bands showing a preferred orientation in a matrix of interlocked equigranular quartz grains displaying strongly irregular grain boundaries. Large euhedral porphyroblasts of garnet, forming one of the major phases, are dispersed throughout the rock.

Accessory subhedral tabular and rhombic sections of actinolite (colourless to bluish green pleochroism) occur orientated parallel to the schistosity. The actinolite also occurs as rims to euhedral grains of rhombic and tabular outline which may have originally been brown hornblende but now are pseudomorphed by what appears to be a mixture of talc, chlorite and residual hornblende. Some talc is present as small pockets within the muscovite layers but this identification is based on the form, the lower refractive index and the occurrence of dusty inclusions. The colour, birefringence etc. of the talc is otherwise the same as muscovite.

In polished section the main opaque accessory mineral is pyrrhotite occurring as subhedral laths lying parallel to the schistosity. Traces of chalcopyrite also occur, and some rutile rods mainly as inclusions in the garnet porphyroblasts.



Fig. 4a Photomicrograph of polished section of I7 showing pyrrhotite (white), garnet (light grey), and muscovite-quartz (darker grey). Very dark to black areas are pits in the surface.

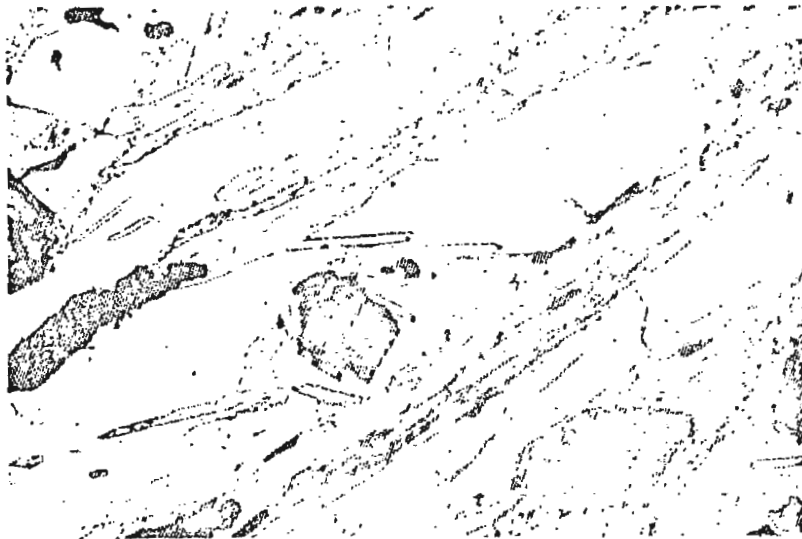


Fig. 4b. Photomicrograph, mag. x 40, of thin section of I7 consisting of garnet, muscovite and quartz under plane polarised light.

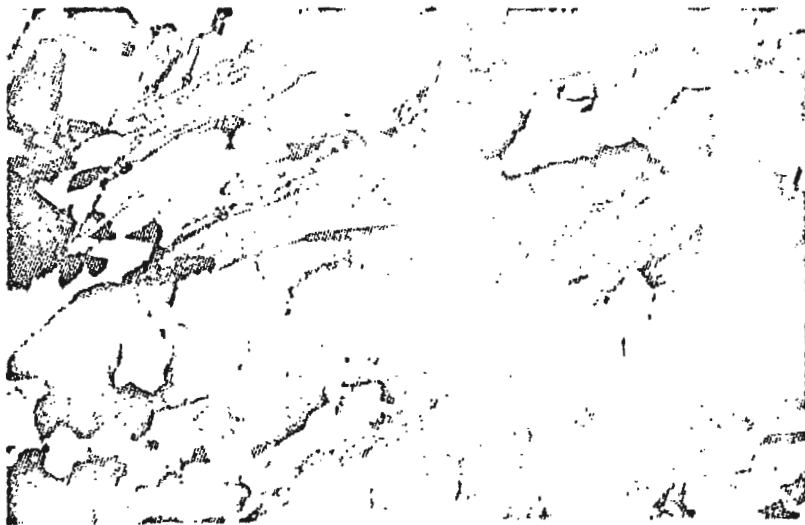


Fig. 4c. Photomicrograph, mag. x 40, of thin section of I7 under crossed nicols showing subhedral garnet (black), anhedral interlocking quartz (white-grey-black), and lamellar muscovite (coloured).

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Specimen I8

In hand specimen I8 appears as a coarse aggregate of foliaceous talc varying in colour from white to greenish white. The general texture in thin section is of coarse foliated talc preferentially orientated and alternating with long lenses of a finer talc in which a preferred orientation appears to be absent as a result of shearing parallel to the schistosity. Minor chlorite (var. sheridanite) occurs as orientated laths intimately intergrown with the coarse talc, and as fibrous aggregates in the finer talc lenses. Rare anhedral garnet, possibly pyrope, occurs.

In thin section the talc which appears greenish in hand specimen is seen to be crowded with minute inclusions of a pinkish mineral occurring as rounded to thin tabular grains and having a lower refractive index than the talc. A grey-brown amorphous material is also present. This material together with the granular inclusions is presumably responsible for the greenish colouration of the talc in hand specimen. As in I3 and I5 the greenish talc has been cleansed of inclusions along planes parallel to the schistosity by some later metasomatic process or retrograde metamorphic process. This 'absorption' of the inclusions by the talc or removal of the inclusions does not effect the form of aggregation of the talc crystals. Boundaries between the clean transparent and 'murky' talc often transgress the schistosity and there is no change in the coarseness or mode of aggregation of the talc across such boundaries. X-ray diffraction of the transparent white talc and the translucent greenish talc revealed no differences and the composition of these inclusions is at the moment unknown. Figure 5, under crossed nicols, shows such a transgressive boundary between the clear and 'murky' or dusty talc.

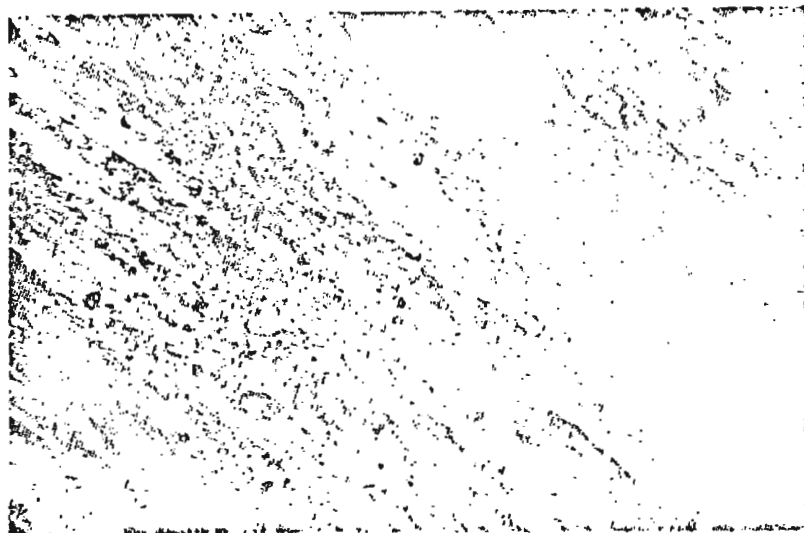


Fig. 5. Photomicrograph, mag x 24, of thin section I8 showing the nature of the talc intergrowth under crossed nicols, and the transgressive boundaries between clear transparent talc and the inclusion-rich 'murky' talc which appears greenish white in hand specimen.

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Specimen I₉: 'Grey talc 1st face'.

In specimen I₉ talc and chlorite (var. sheridanite) are the main constituents. They occur intimately intergrown as long orientated foliaceous aggregates alternating with finer platy aggregates in which the talc and chlorite fibres are randomly orientated and which form lenses elongated parallel to the schistosity of the coarser foliaceous talc (Figs. 6a and 6b). As in previous sections the talc appears murky in parts due to the presence of minute unidentifiable inclusions.

The talc is also crowded with small irregular and rod-shaped grains of rutile. Rare subhedral porphyroblasts of garnet (possibly pyrope) also occur.

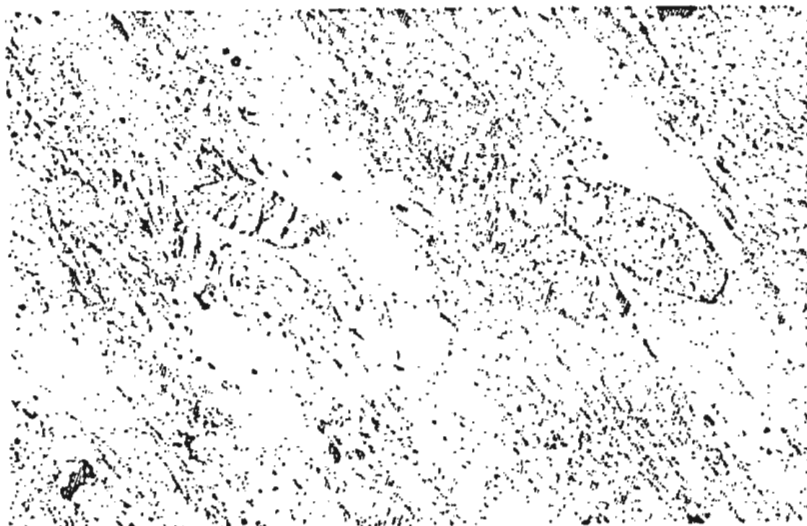


Fig. 6a Photomicrograph, x 40 mag, of thin section I₉ under plane polarised light showing subhedral garnet grains in an orientated foliaceous aggregate of talc and chlorite.

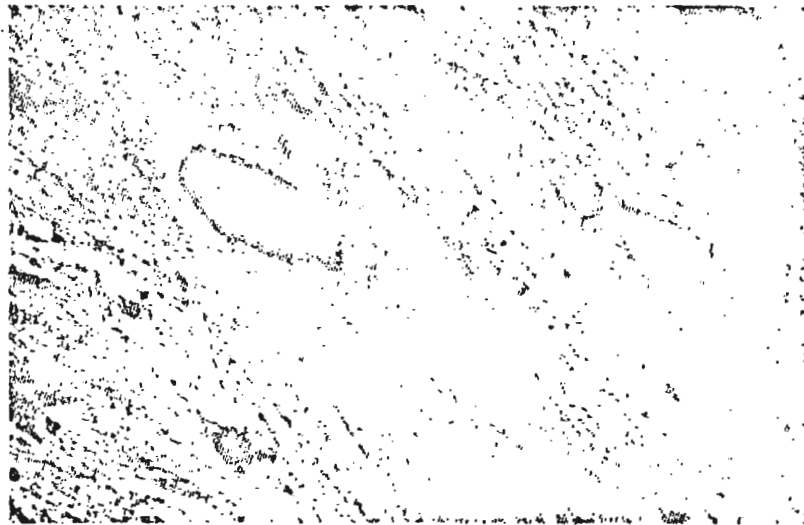


Fig. 6b. Photomicrograph, x 40 mag., of thin section I₉ under crossed nicols showing garnet (black) in a coarse matrix of foliaceous talc (bright interference colours) and chlorite (white to blue-grey interference colours).

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Specimen I₁₀ and I_{10A} : 'granular talc'

Both I₁₀ and I_{10A} consist of an intergrowth of medium grained and randomly orientated major talc with minor chlorite (var. sheridanite) (Fig. 7). Some small porphyroblasts of garnet also occur scattered in the talc/chlorite ground mass. In this specimen the talc is not crowded with inclusions as is the case in most of the other samples.

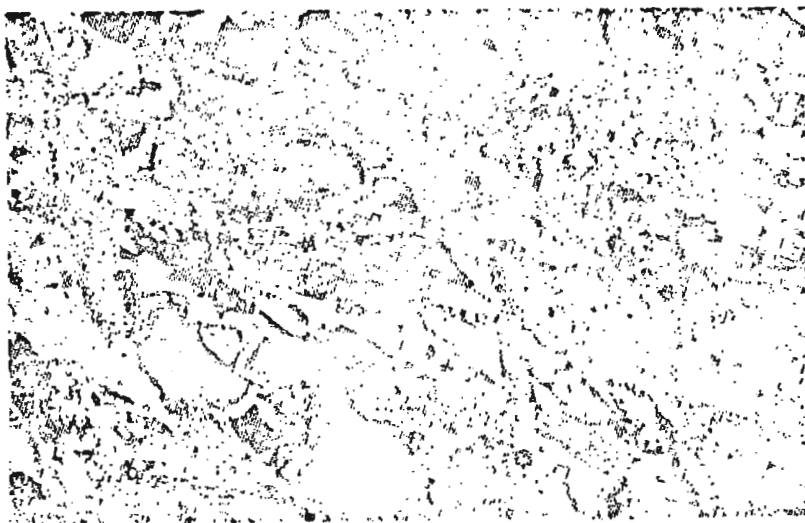


Fig. 7. Photomicrograph, x 40 mag., of thin section I₁₀, under crossed nicols, consisting of talc (blue and yellow interference colours), chlorite (white and greys), and garnet (black).

Specimen I₁₁ : 'carbonate and talc'

Specimen I₁₁ consists dominantly of a mosaic of coarse to fine grained anhedral interlocking magnesite grains with interstitial pockets of coarse to medium grained foliaceous aggregates of talc (Figs. 8a and 8b). The talc is crowded with near sub-microscopic inclusions of a transparent phase together with a brown amorphous material which causes the talc to appear dusty or turbid in thin section. Some fibrous chlorite (var. sheridanite) occurs as small pockets intergrown with the talc. Traces of rutile and pyrite occur.



Fig. 8a. Photomicrograph, x 24 mag., of thin section I₁₁ under plane polarised light showing a subhedra pyrite metacryst (black) in a matrix of compact granular magnesite with interstitial foliaceous talc (top centre).

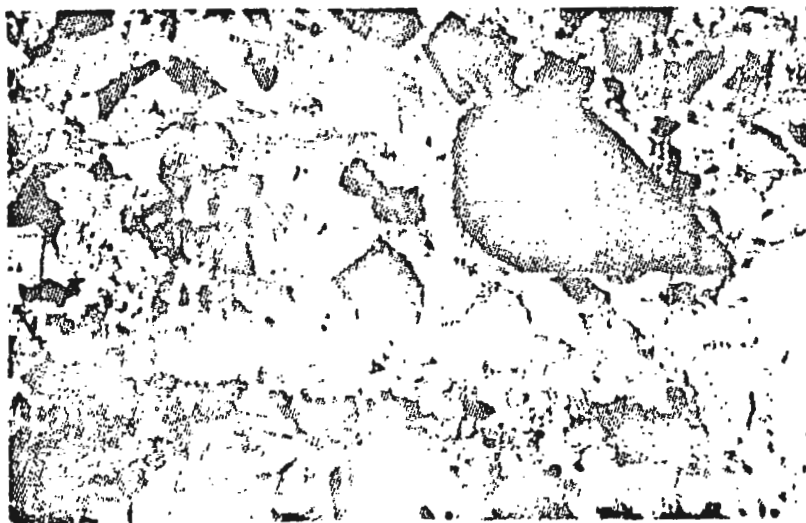


Fig. 8b. Photomicrograph, x 24 mag., of thin section I₁₁ under crossed nicols showing a pyrite metacryst (black) in a granular magnesite matrix, with a foliaceous interstitial aggregate of talc (top centre).

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Specimen I₁₂

An aggregate of anhedral quartz as the main constituent with minor interstitial muscovite and green chlorite (var. pennine) Fig. 9. The long muscovite laths show a preferred orientation. Chlorite occurs in interstitial pockets as randomly orientated platy grains. Some epidote is present and a trace of magnesite.

The chlorite displays a pleochroism from light green to brownish-cream, and anomalous blue interference colours in some cases. However, most of the chlorite grains display lower second order to upper first order interference colours. Thus a range of chlorite composition is probably represented in the section.

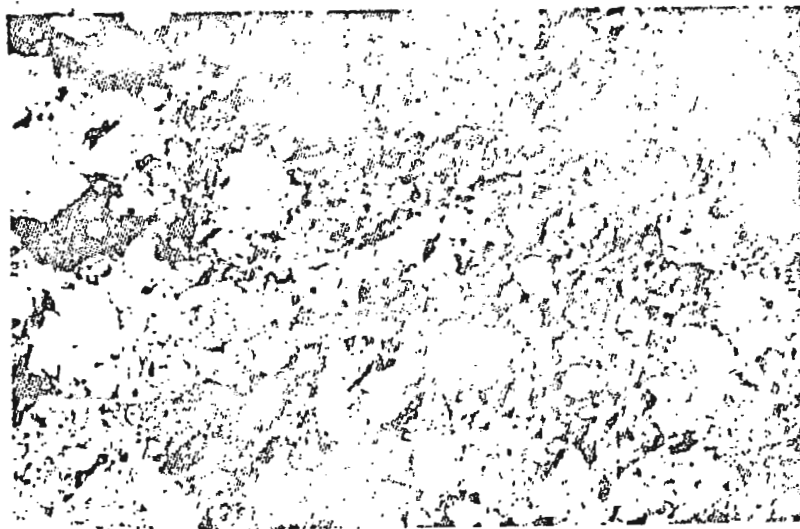


Fig. 9. Photomicrograph, x 40 mag., of thin section I₁₂ under crossed nicols.

Specimen I₁₃

This specimen consists of an aggregate of mainly medium grained platy to fibrous chlorite (var. sheridanite) and equigranular quartz. These two enclose ragged replacement residuals of calcite and subhedral metacrysts of pyrite with rare chalcopryite.

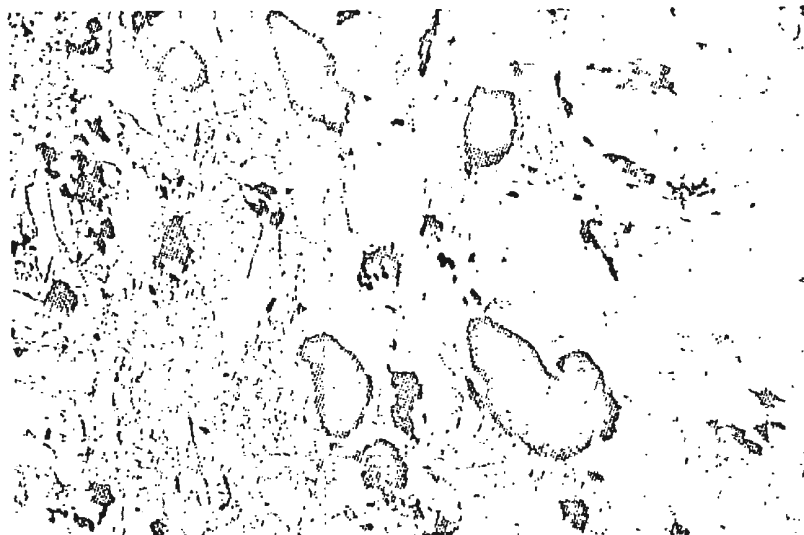


Fig. 10a Photomicrograph, x 40 mag., of thin section I₁₃ under PPL showing subhedral pyrite meta-crystals (black) in a matrix of dominantly chlorite and quartz with minor calcite.

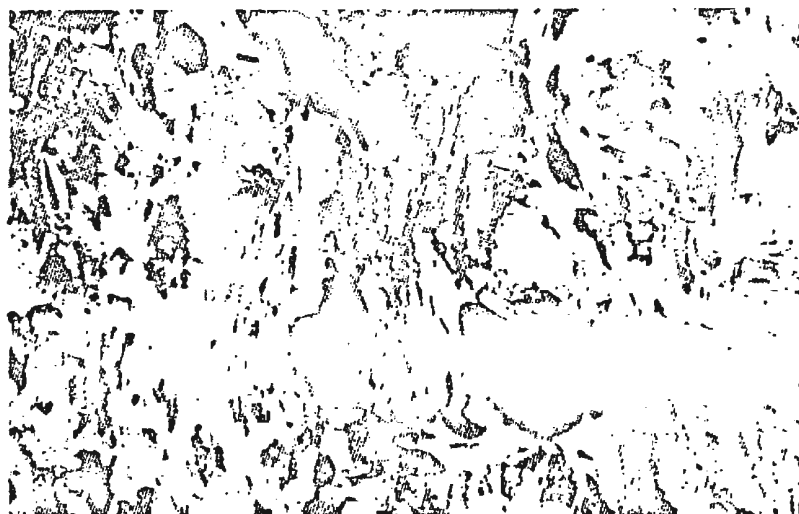
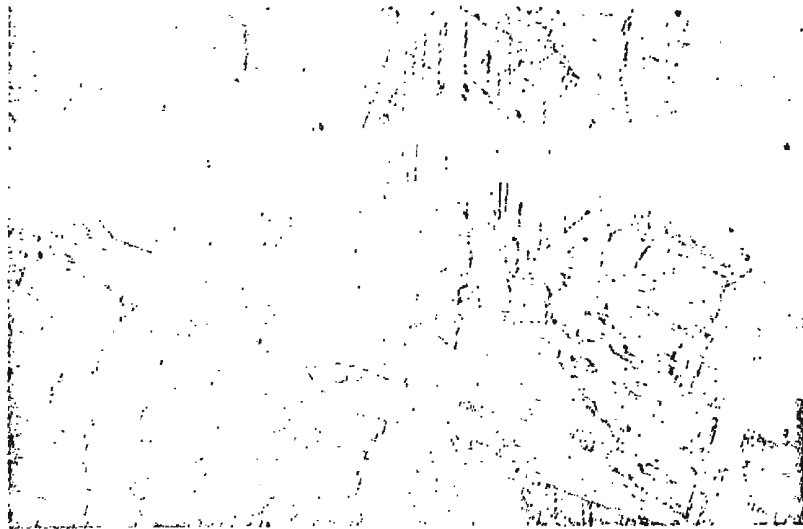


Fig. 10b Photomicrograph, x 40 mag., of thin section I₁₃ under XN showing chlorite (fibrous white and greenish-grey) and calcite (coloured) enclosing subhedral grains of pyrite (black). ~~Quartz grains are quartz.~~

J&J-0130557

Specimen I₁₄

This specimen is dominantly composed of very coarse grained magnesite enclosing minor amounts of talc and very minor chlorite (var. sheridanite). The talc and chlorite form pockets of radiating lamellar and foliaceous crystals as in Figs. 11a, 11b.

Fig. 11a

Photomicrograph, x 24 mag., of thin section I₁₄ under PPL of coarse magnesite and intergranular pockets of 'dusty' and 'clear' talc.

Fig. 11b

Photomicrograph, x 24 mag., of thin section I₁₄ under XN of magnesite (greenish) and pockets of radiating lamellar talc (blue, purple, yellow).

J&J-0130558

Specimen I15A

This specimen of wallrock is a garnet-muscovite-quartz schist with minor green chlorite, biotite, and rare talc and feldspar (Figs. 12a and 12b).

The garnet occurs as large (1-3mm diam.) porphyroblasts altered along irregular fractures to a mixture of greenish chlorite, biotite, and some feldspar, and enclosed in a matrix composed of orientated tabular grains of muscovite, forming elongated lenses, and alternating with 'mosaic' granular quartz containing randomly dispersed biotite and chlorite flakes.

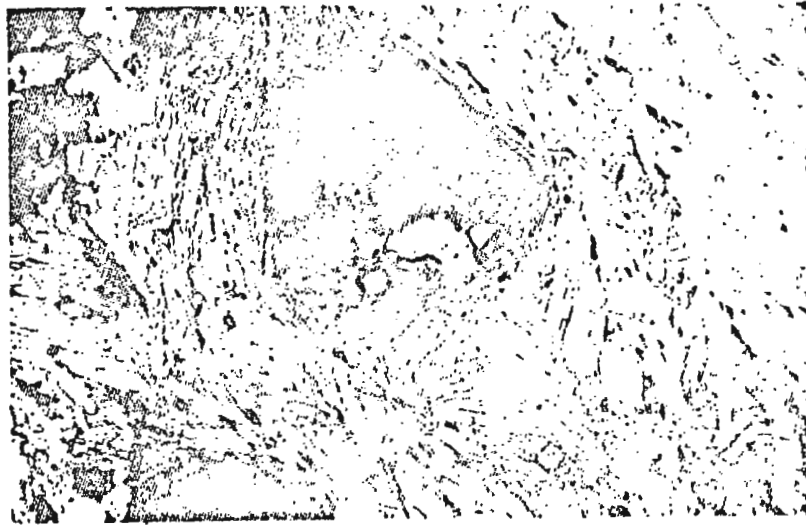


Fig. 12a Photomicrograph, x 24 mag., of thin section I15A UNDER PPL showing a large altered porphyroblast of garnet in a matrix of dominantly muscovite with minor quartz.

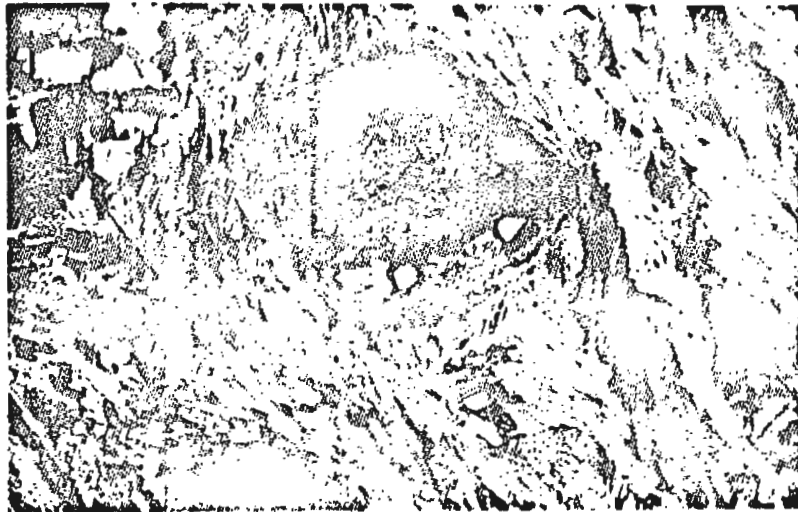


Fig. 12b. Photomicrograph, x 24 mag., of thin section I_{15A} under crossed nicols. Garnet (black). Muscovite (dominantly purple interference colours). Quartz (white and greys).

Specimen I₁₅

This specimen is dominantly composed of chlorite (var. sheridanite) and quartz as orientated aggregates producing a schistosity. Very minor amounts of magnesite and talc occur. The talc occurs as thin laths intergrown with the chlorite (Fig. 13b).



Fig. 13a Photomicrograph, x40 mag., of thin section I₁₅ under PPL showing the irregular but preferred elongation of granular quartz segregations in a matrix of fibrous chlorite (var. sheridanite).

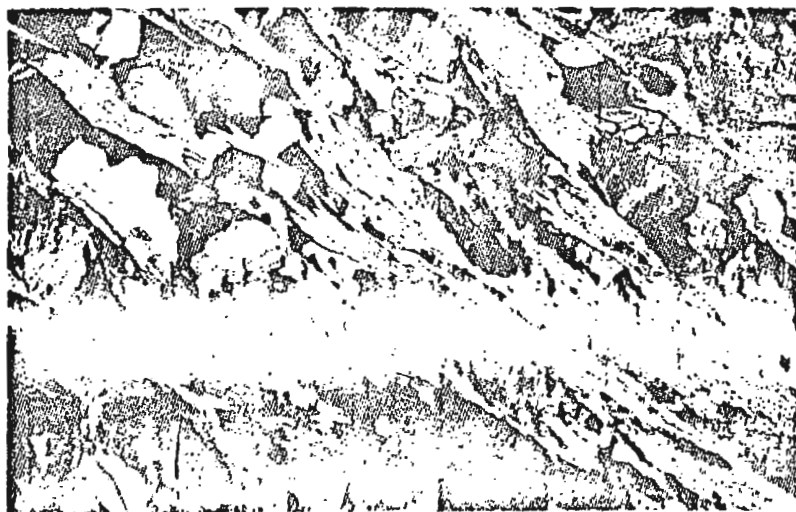


Fig. 13b Photomicrograph, x 40 mag., of thin section I₁₅ under XN, composed of chlorite (fibrous white, greenish grey, black), quartz (granular white-grey-black), and talc (blue, red, and yellow interference colours).

J&J-0130561

JNJAZ55_000017581

Specimen I16: 'first face inclusion'

480

This specimen is composed of a medium grained aggregate of dominantly chlorite (var. sheridanite) and quartz, with minor magnesite, clinozoisite, talc, and muscovite, and displaying a poor schistosity. Scattered euhedral to subhedral pyrite metacrysts occur as well as medium grained crystal aggregates of rutile associated with clinozoisite forming 'stringers' parallel to the general schistosity of the rock.

In the photomicrograph of figure 14a the brownish speckled areas are dominantly chlorite although in Figure 14b talc and muscovite are more apparent because of their interference colours.

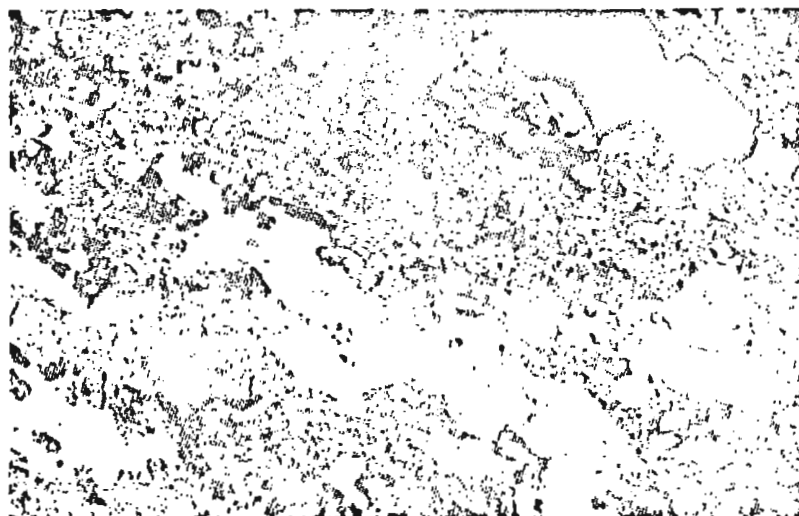


Figure 14a Photomicrograph, x 40 mag., of thin section I16 under PPL.

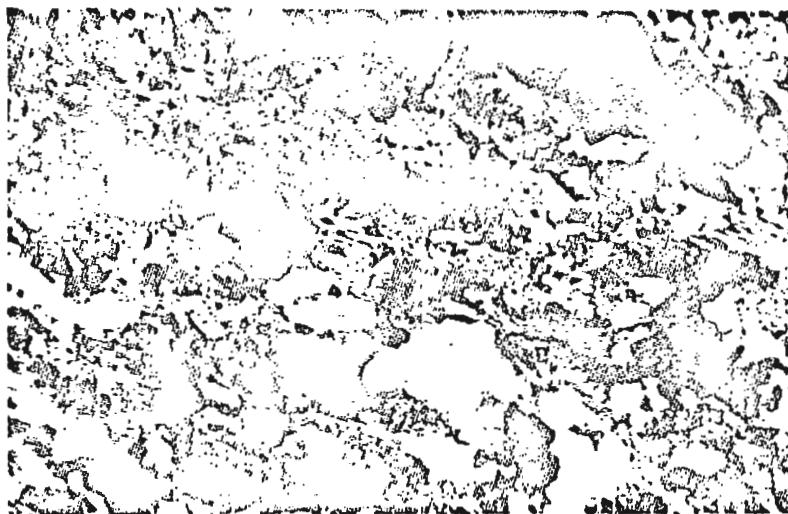


Fig. 14b Photomicrograph, x 40 mag., of thin section I₁₆ under crossed nicols. A chlorite - quartz rock with minor talc and muscovite, and accessory magnesite, clinozoisite, rutile and pyrite.

J&J-0130563

Specimen I₁₇: 'footwall'

This specimen of footwall rock is a muscovite-quartz-garnet schist consisting of long lenticular anhedral quartz aggregates. Both are enclosing fractured and altered euhedral porphyroblasts of garnet. Accessory sphene also occurs as well as serpentine-quartz pseudomorphs after a mineral displaying rhombic and tabular sections.



Fig. 15a Photomicrograph, x 24 mag., of thin section I₁₇ under PPL showing garnet euhedra in a matrix of segregated quartz and muscovite.

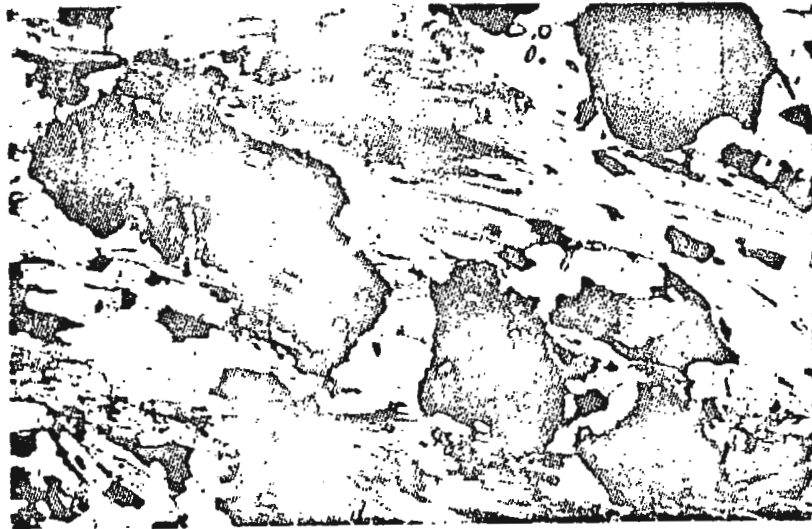


Fig. 15b Photomicrograph, x 24 mag., of thin section I₁₇ under XN. Garnet (black), quartz (white to grey), and muscovite (lamellar and coloured).

J&J-0130564

JNJAZ55_000017584

Specimen I₁₈: 'Face 3, carbonate/talc'

A coarse to medium grained aggregate of subhedral interlocking grains of magnesite with minor talc occurring as scattered small interstitial clusters associated with rare chlorite (var. sheridanite) and muscovite (Figs. 16a, 16b).

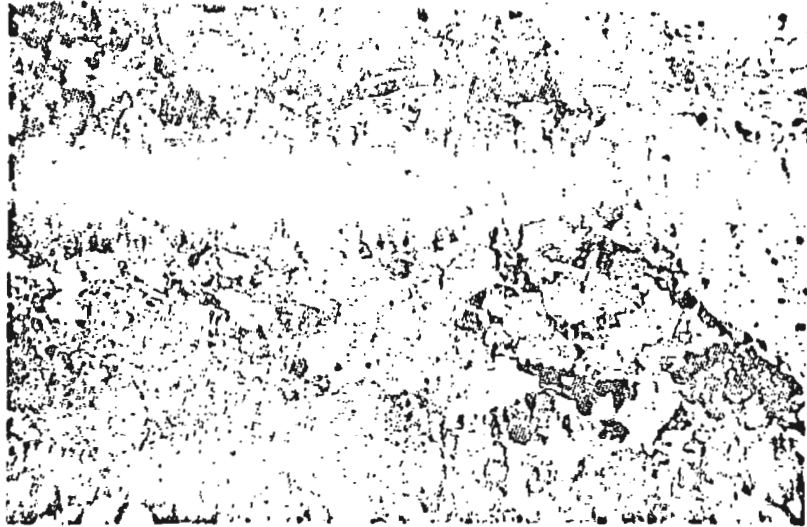


Fig. 16a Photomicrograph, x 24 mag., of thin section I₁₈ under PPL of granular magnesite with scattered tabular crystals and clusters of talc.

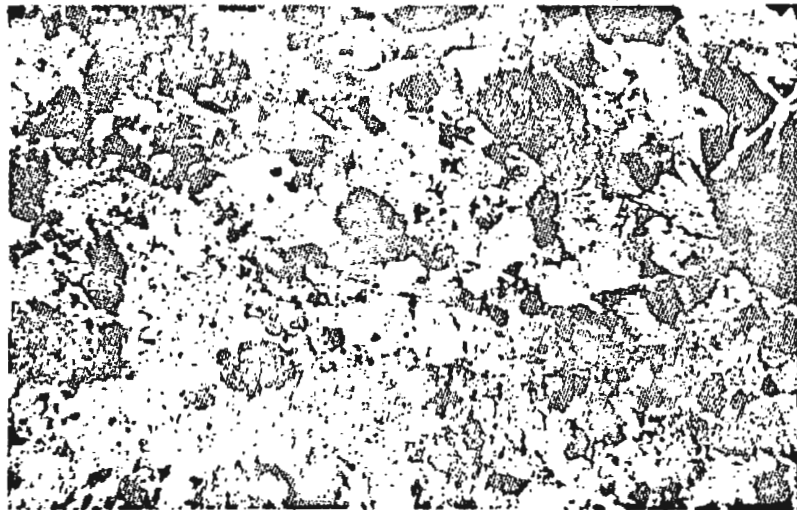


Fig. 16b Photomicrograph, x 24 mag., of thin section I₁₈ under XN of granular magnesite (high order interference colours, and scattered tabular crystals and clusters of talc (top right, coloured) and rare chlorite (white to blue-grey colours)).

Specimen I₁₉:

This specimen consists of an aggregate of coarse grained anhedral magnesite intergrown with solitary bladed crystals and crystal aggregates of tremolite associated with minor amounts of fine fibrous talc and rare anhedral grains of quartz (Figs. 17a, 17b).



Fig. 17a Photomicrograph, x 24 mag., of thin section of I₁₉ under PPL, showing coarse bladed tremolite intergrown with very coarse grained magnesite.

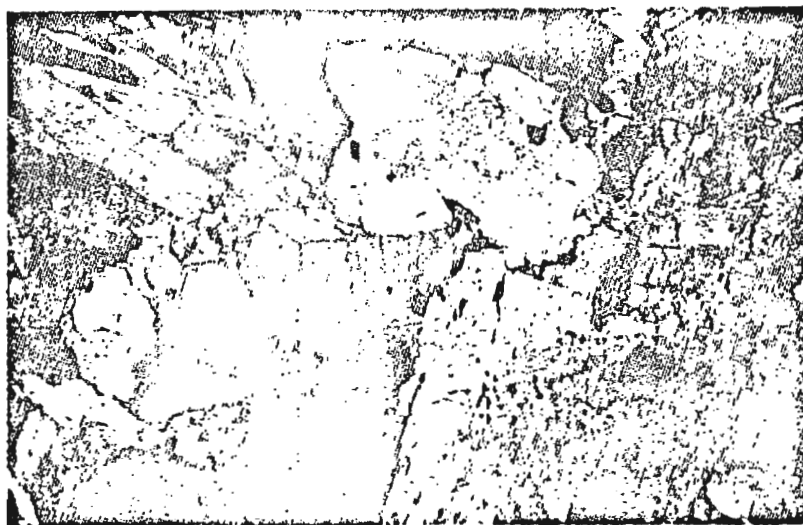


Fig. 17b Photomicrograph, x 24 mag., of thin section I₁₉ under crossed nicols showing coarse bladed tremolite and anhedral coarse-grained magnesite with minor small fibrous aggregates of talc (top left)..

J&J-0130566

Specimen I21: 'Inclusion, face 2'.

Specimen I21 is composed of a fine grained interlocking aggregate of anhedral magnesite, as the major constituent, associated with scattered laths and interstitial fine-grained fibrous aggregates of very minor talc (Figs. 18a and 18b).

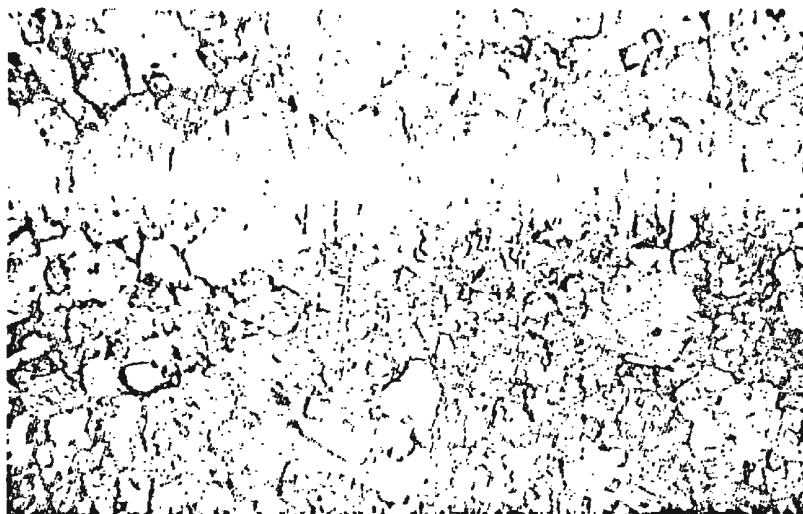


Fig. 18a Photomicrograph, x 24 mag., of thin section I21 under PPL. Magnesite with rare talc.

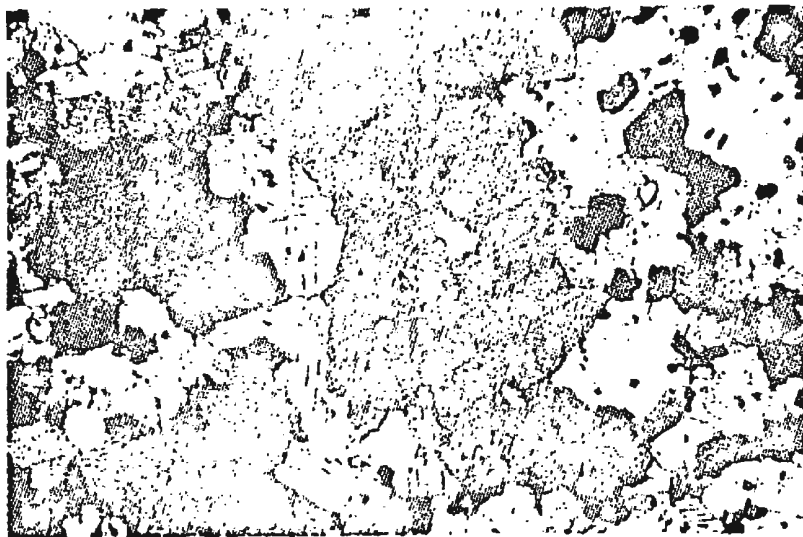


Fig. 18b Photomicrograph, x 24 mag., of thin section I21 under crossed nicols. Magnesite with rare talc.

Specimen I22

This specimen is dominantly composed of coarse subhedral to euhedral interlocking grains of magnesite associated with intergranular fibrous clusters of talc which often enclose smaller euhedral magnesite grains (Fig. 19).



Fig. 19 Photomicrograph, x 24 magnification, of thin section I22 under plane polarised light. Magnesite and interstitial aggregates of talc.

Specimen I23: 'Black Gneiss 2' below talc vein'

Specimen I23 consists dominantly of medium grained anhedral interlocking quartz as orientated bands enclosing large microcline anhedral and anhedral aggregates. Scattered platy aggregates of muscovite occur orientated parallel to the general direction of the quartz banding. Minor epidote and chlorite also occur (Figs. 20a and 20b).

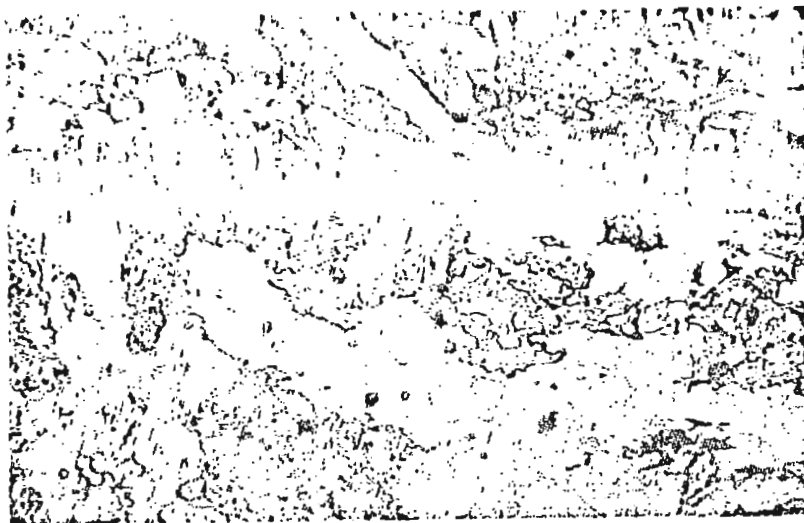


Fig. 20a Photomicrograph, x 24 mag., of thin section I23 under PPL. Quartz-muscovite-microcline gneiss.

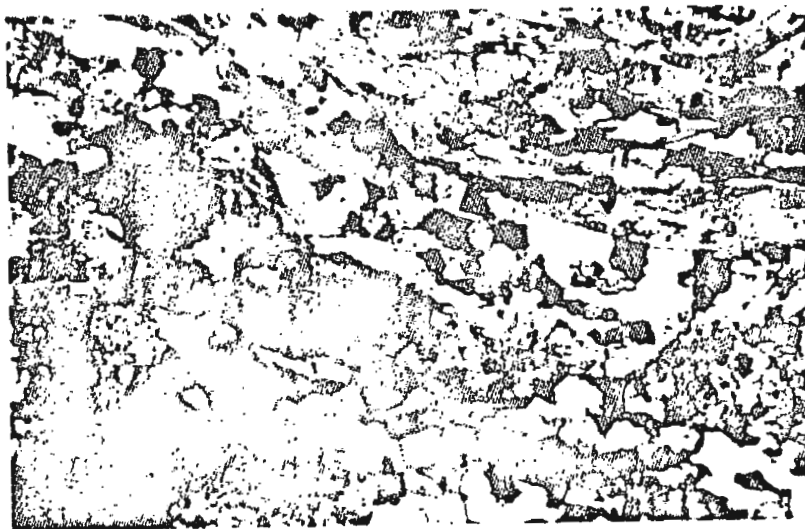


Fig. 20b Photomicrograph, x 24 mag., of thin section I23 under XN. Quartz-muscovite-microcline gneiss.

Specimen I₂₄: 'Face 2, Talc next to carbonate'

This specimen of talc ore consists dominantly of coarse fibrous talc with minor chlorite (var. sheridanite) occurring as small lenticular fibrous aggregates within the main mass of talc (Figs. 21a and 21b). A few small subhedra of garnet are present. As in previous specimens there are two forms of talc present: (1) a talc that in thin section appears brown (Fig. 21a) under plane polarised light due to finely dispersed dusty inclusions of a transparent mineral and a brown amorphous material, (2) a clear transparent talc free of inclusions which appears to have been formed at the expense of the other by some metasomatic 'cleansing' process. Talc crystals in optical continuity can be seen to change sharply from 'dusty' brown talc to the clear talc.

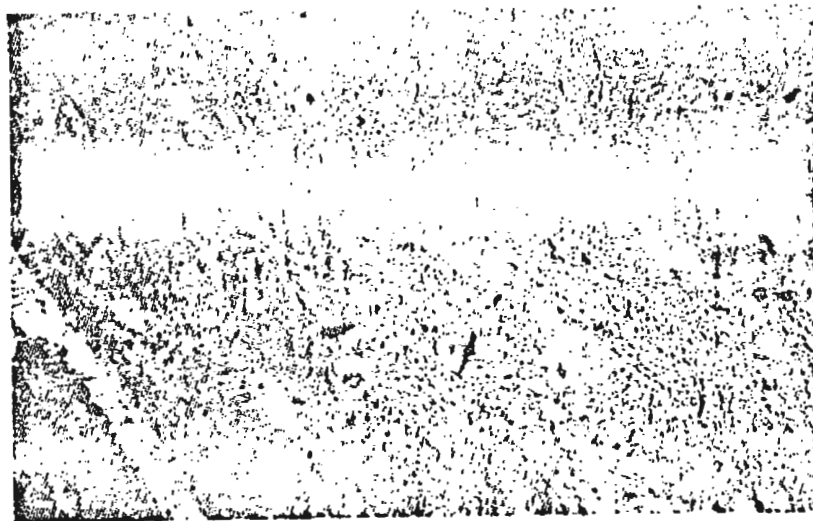


Fig. 21a Photomicrograph, x 24 mag., of thin section I₂₄ under PPL. 'Dusty' and clear talc enclosing small lenticular aggregates of chlorite.

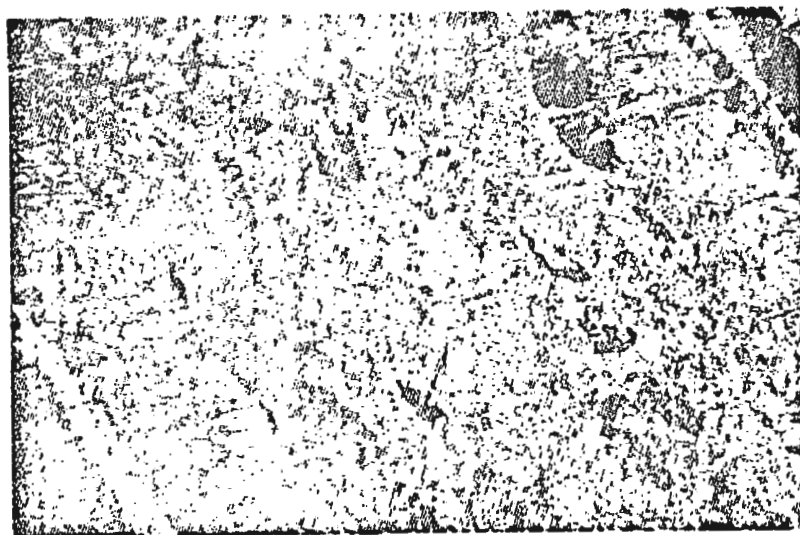


Fig. 21b Photomicrograph, x24 mag., of thin section I₂₄ under XN. Coarse talc with lenticular aggregates of chlorite.

J&J-0130570

JNJAZ55_000017590

This specimen of footwall rock consists of an interlocking aggregate of medium grained anhedral quartz enclosing occasional large anhedral microcline feldspar (Figs. 22a, 22b). Minor magnesite occurs as pockets interstitial to the quartz, and also scattered laths of muscovite. Green chlorite (pennine) and epidote occur in trace amounts.

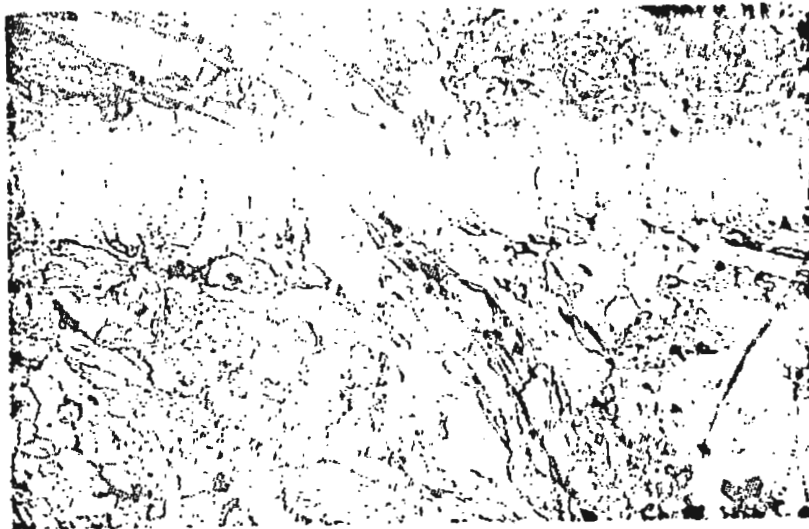


Fig. 22a Photomicrograph, x 24 mag., of thin section I25 under PPL; dominantly a quartz-microcline rock with minor muscovite and rare pennine and epidote.

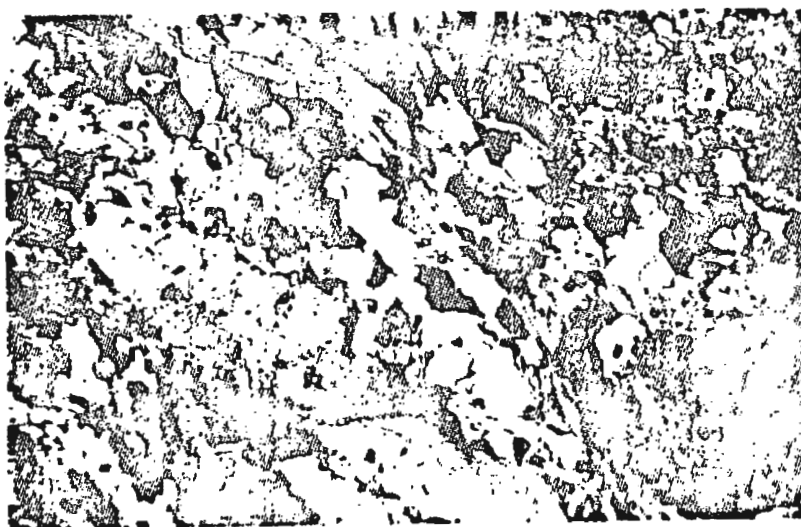


Fig. 22b Photomicrograph, x 24 mag., of thin section I25 under XN.

Specimen I26

This specimen contains chlorite, talc, magnesite and rutile. One part of the thin section consisted of a massive coarse fibrous and feathery aggregate of talc enclosing pockets of coarse magnesite. This texture graded into one which was dominantly fine grained chlorite (var. sheridanite) intimately intergrown with minor quantities of fibrous and platy talc (Fig. 23) as well as scattered small equigranular and rod-shaped rutile crystals.



Fig. 23. Photomicrograph, x 40 mag., of thin section I26 under crossed nicols showing minor talc (coloured) intimately intergrown with major chlorite.

Specimen I27

Specimen I27 is dominantly composed of quartz, chlorite (var. sheridanite) and talc (Figs. 24a and 24b). Thin lenticular bands of coarse feathery talc and chlorite alternate with anhedral granular interlocking aggregates of quartz. Scattered inclusions of rutile and epidote occur, as well as occasional large microcline anhedral.

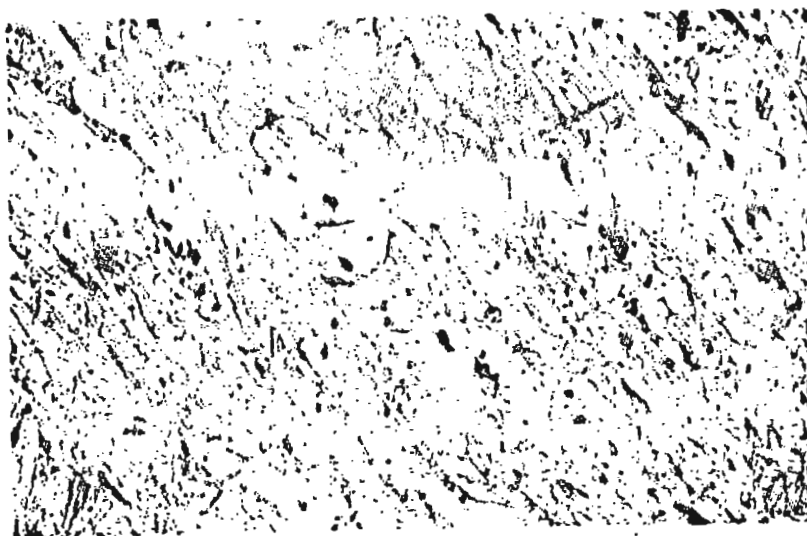


Fig. 24a Photomicrograph, x 40 mag., of thin section I27 under PPL, showing a fibrous and feathery aggregate of talc and chlorite enclosing anhedral segregations of quartz.

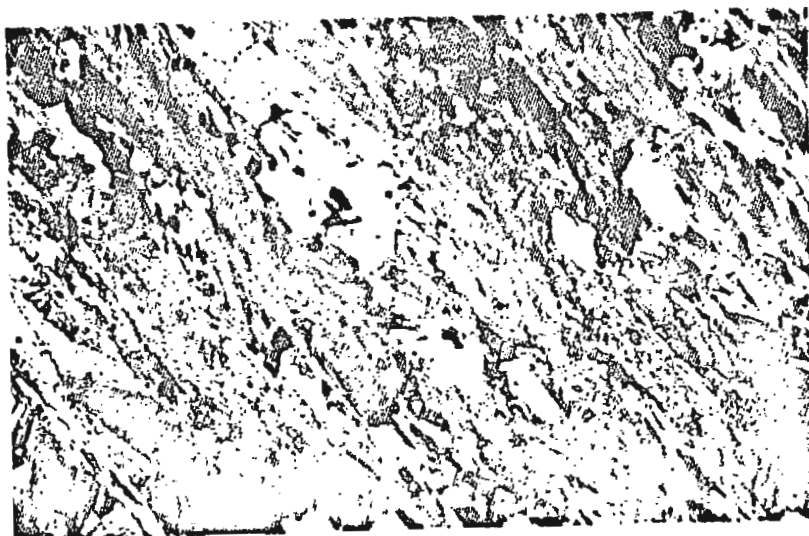


Fig. 24b Photomicrograph, x 40 mag., of thin section I27 under XN.

J&J-0130573

JNJAZ55_00001759

Specimen I29

Specimen I29 is a gneissic rock consisting of segregated bands of medium to fine interlocking anhedral quartz grains alternating with minor muscovite as orientated platy clusters and enclosing large microcline anheda. Some rare pennine and very rare epidote occur intergrown with the muscovite.



Fig. 25a Photomicrograph, x 24 mag., of thin section I29 under PPL; quartz, muscovite, and microcline (top left)



Fig. 25b Photomicrograph, x 24 mag., of thin section I29 under XN.

Specimen I31

Specimen I31 is a muscovite-quartz schist containing minor pennine, sphene and tremolite.

The rock is dominantly made up of coarse orientated lamellar segregations of muscovite intergrown with flakes of minor greenish brown chlorite (pennine) and enclosing euhedral to subhedral grains of sphene. Minor interlocking fine to medium grained quartz segregations occur alternating with the muscovite bands. Hexagonal sections of an amphibole, probably tremolite, occur dispersed in the muscovite matrix.

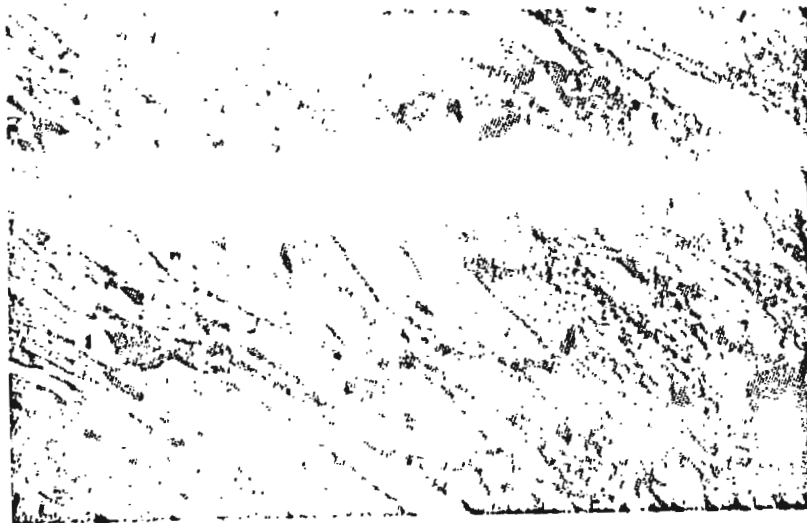


Fig. 26a Photomicrograph, x 40 mag., of thin section I31 under PPL; muscovite-quartz schist.

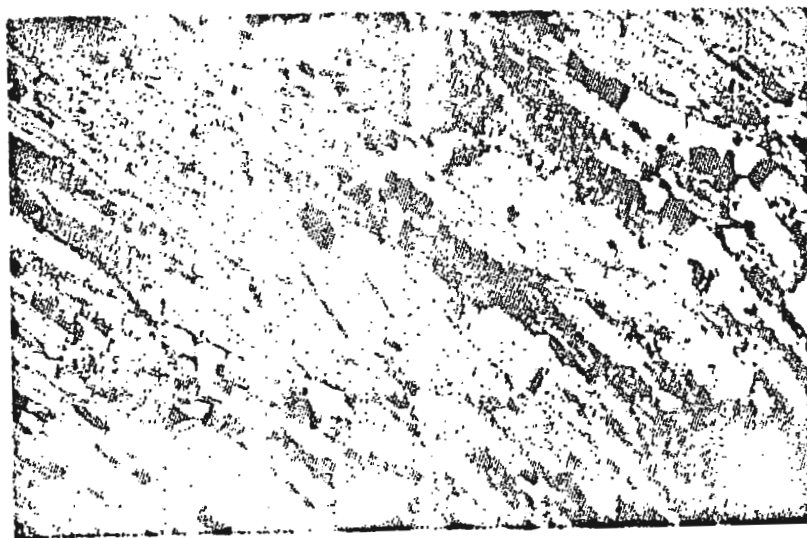


Fig. 26b Photomicrograph, x 40 mag., of thin section I31 under XN; muscovite-quartz schist.

J&J-0130575

JNJAZ55_00001759

Specimen I32

This specimen consists of coarse feathery lenticular aggregates of dominantly chlorite (var. sheridanite) intimately intergrown with minor amounts of talc (Figs. 27a and 27b).

Small inclusions of rutile occur along the boundaries (shear planes) between the chlorite aggregates and also along chlorite cleavage planes. Finely dispersed submicroscopic dusty inclusions of an unidentified phase similar to that found in talc occur in the chlorite.



Fig. 27a Photomicrograph, x 24 mag., of thin section I32 under XN. Feathery aggregates of sheared chlorite (white to greenish grey to black) with minor talc (coloured).

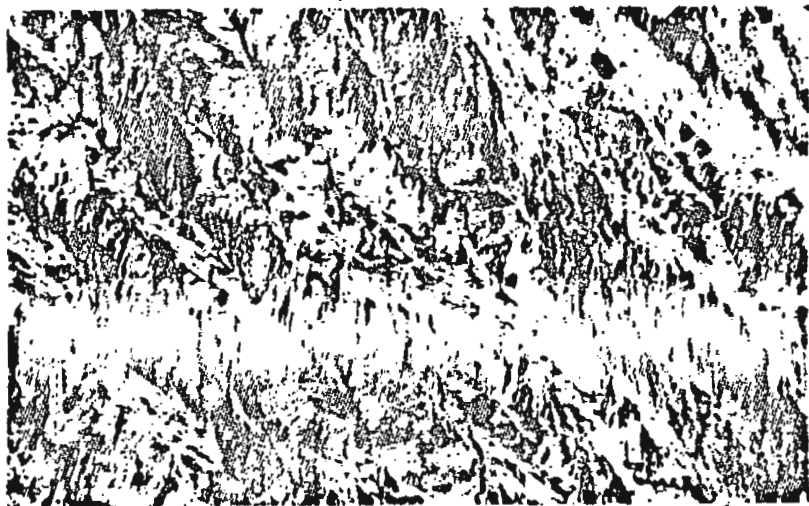


Fig. 27b Photomicrograph, x 24 mag., of thin section I32 under XN. Finer grained chlorite-talc mixture.

J&J-0130576

JNJAZ55_000017596

Specimen I33

This specimen of talc ore consists of a medium to fine grained randomly orientated intergrowth of dominantly talc with minor chlorite (var. sheridanite). The chlorite is intimately mixed with the talc (Fig. 28). Some pockets of coarse interlocking anhedral magnesite grains occur enclosed by the talc-chlorite matrix.



Fig. 28 Photomicrograph, x 24 mag., of thin section I33 under XN.

Specimen I35

This specimen consists dominantly of magnesite as a very coarse to medium grained interlocking aggregate of euhedral to subhedral grains. Minor tremolite occurs as long prismatic crystals forming interstitial clusters, and as solitary crystals penetrating the magnesite and along the grain boundaries of the magnesite. Minor chlorite (var. sheridanite) and rare talc occur associated with the tremolite segregations. (Figs. 29a, 29b).

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Fig. 29a Photomicrograph, x 24 mag., of thin section I35 under PPL. Magnesite-tremolite-chlorite-talc rock.

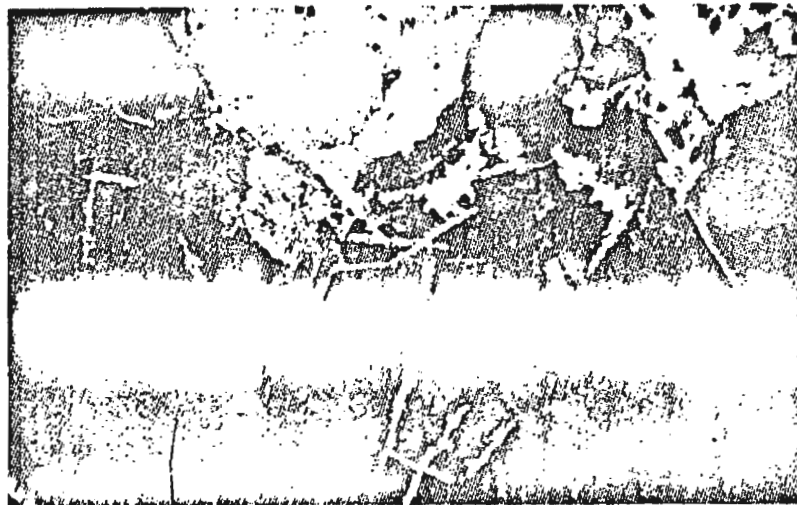


Fig. 29b Photomicrograph, x 24 mag., of thin section I35 under XN. Prismatic tremolite in magnesite in the extinction position.

J&J-0130578

Specimen I37

This specimen consists dominantly of magnesite with minor talc. The magnesite occurs as an aggregate of very large magnesite anhedral enclosed by finer grained subhedral magnesite which is intergrown with feathery intergranular clusters of talc (Fig. 30).



Fig. 30 Photomicrograph of thin section I37, x 24 mag., under XN showing the finer intergranular magnesite associated with small laths of talc (fibrous and coloured).

Specimen I39

This specimen is dominantly composed of talc forming coarse feathery aggregates intimately intergrown with minor finer grained chlorite (var. sheridanite) and containing fine disseminated inclusions of rutile. Occasional fine grained quartz as well as larger oval-shaped augen of quartz and rare garnet occur scattered throughout the talc matrix. The talc is for the most part crowded with inclusions, as in previous sections, but elongate areas of 'clean' talc occur as in Fig. 31a.

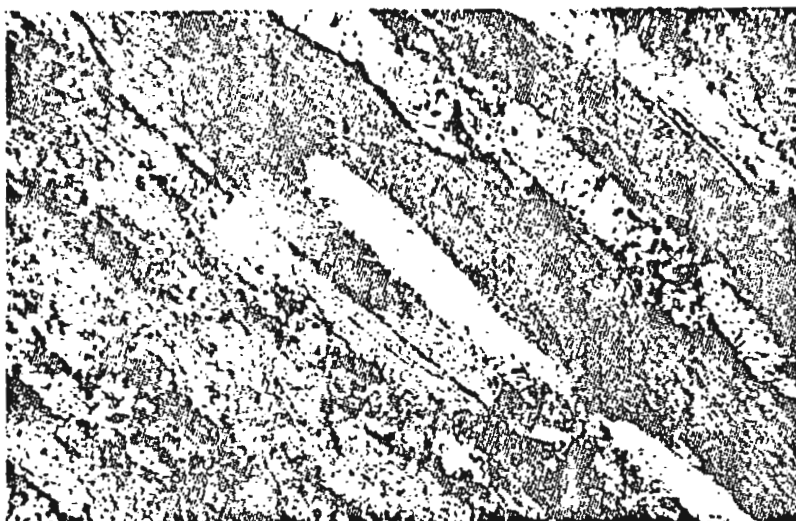


Fig. 31a Photomicrograph, x 24 mag., of thin section I39 under PPL.

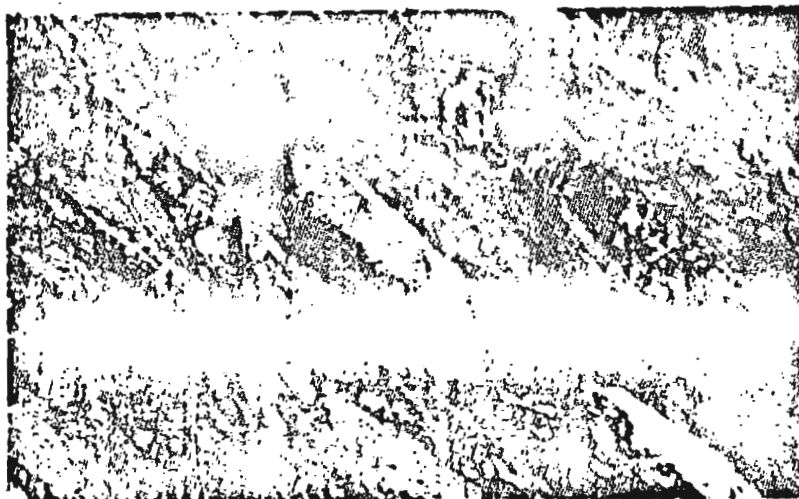


Fig. 31b Photomicrograph, x 24 mag., of thin section I39, under XN

J&J-0130580

JNJAZ55_000017600

Specimen I41

This specimen of talc ore consists of a coarse aggregate of feathery talc intimately intergrown with minor chlorite (var. sheridanite), and enclosing rare large porphyroblasts of subhedral garnet which occasionally contain long prismatic inclusions of tremolite (Fig.32a).



Fig. 32a Photomicrograph, x 24 mag., of thin section I41 under XN. Feathery aggregate of talc with garnet porphyroblast (bottom right, black).

J&J-0130581

JNJAZ55_000017601

Specimen I42: 'No.1 Face, green coloured'

Specimen I42 consists dominantly of an aggregate of fine grained fibrous chlorite (var. sheridanite) intimately intergrown with minor very fine grained talc as in Fig. 33.



Fig. 33 Photomicrograph, x 24 mag., of thin section I42 under crossed nicols of chlorite (white, greenish grey, black), and fine grained talc (yellow).

J&J-0130582

JNJAZ55_000017602

Specimen I43: 'Face 10 fibrous sample'

Specimen I43 consists dominantly of chlorite (var. sheridanite), occurring in the form of a coarse sheared fibrous aggregate intimately intergrown with very minor talc as in Figure 34.

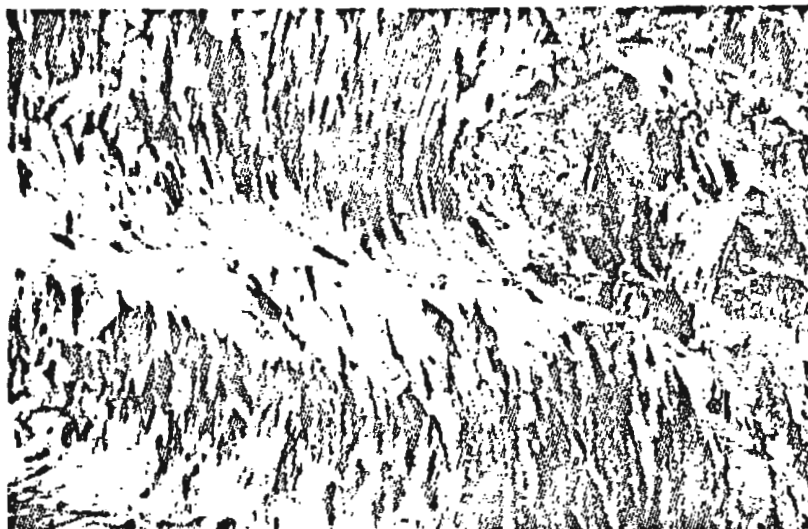


Fig. 34 Photomicrograph, x 40 mag., of thin section I43 under crossed nicols showing deformed fibrous chlorite (white-greenish grey-black) intergrown with platy and prismatic crystals of talc (coloured).

Specimen I43A

As for I43 the specimen consisted dominantly of chlorite (var. sheridanite) with very minor talc. The 'cross fibre' type texture found in I43 and produced by shearing at right angles to the schistosity was absent in specimen I43A.

Specimen I44: 'First face pure talc'

A coarse aggregate of lamellar talc showing a preferred orientation and enclosing augen of what appears to be an intimate intergrowth of quartz and serpentine (Fig. 35). Both talc crowded with fine unidentified inclusions and 'clear' talc are present. See also description for I45.

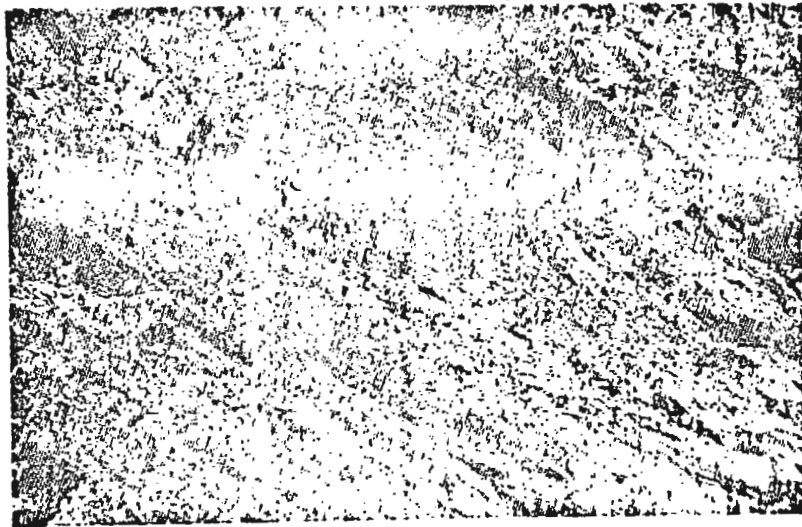


Fig. 35 Photomicrograph, x 24 mag., of section I44 under crossed nicols showing coarse lamellar talc enclosing rare anhedral segregations of probable serpentine-quartz composition.

J&J-0130584

JNJAZ55_000017604

Specimen I45: 'No.1 good specimen' .

This specimen of 'talc ore' consists nearly wholly of talc occurring in the form of a randomly orientated 'matted' aggregate of fibrous talc enclosing minor quartz-serpentine augen. As in previous sections the talc is rendered murky or dusty by fine inclusions of a brown amorphous material and an unidentified transparent phase. In places the talc has been cleansed of these inclusions along zones which appear to be independent of any intergrowth or crystallographic features of the talc (Fig. 36).



Fig. 36 Photomicrograph, x 24 mag., of thin specimen I45 under crossed nicols showing the form of aggregation of the talc and the difference between the 'murky' talc and the linear transgressive zone of 'clear' talc.

J&J-0130585

JNJAZ55_00001760

Specimen I46: 'No.3 face, coloured'

This specimen consists of very coarse lenticular aggregates of long fibrous and feathery talc crystals enclosing rare anhedral porphyroblasts of garnet.

J&J-0130586

JNJAZ55_000017606

DIGESTIVE TESTS

To confirm the presence of acid soluble carbonate material and also to help identify the type of carbonate present in the rock specimens collected, each powder specimen was subjected to a digestive test.

Half gram quantities of each of the powders were treated with normal hydrochloric acid for several hours at approximately 70°C. The residues were reweighed and the filtrates were analysed for their calcium and magnesium content using the EEL, 240 Atomic Absorption Spectrophotometer. The aim of the digestion was not to estimate the total acid soluble fraction only to help establish the carbonate minerals present and to estimate roughly their quantity to help interpret the X-ray powder photographs obtained from the samples.

The results are present under three headings, namely 'Rock Types', 'Carbonate Specimens', and 'Talc Specimens'.

It can be seen that only small quantities of carbonate material are present in the talc specimen group, similarly in the rock specimens with the exception of the marble specimen which is practically 100% calcite. The carbonate group of specimens appear to be mixtures of calcium and magnesium carbonate with a number of specimens being possible dolomites.

ROCK TYPES

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Specimen No.	% Weight Loss	% Calcium	% Magnesium
I1	<0.2%	<0.2%	<0.2%
I7	3.0%	<0.2%	<0.2%
I12	<0.2%	<0.2%	<0.2%
I13	4.2%	1.0%	0.4%
I15	6.0%	<0.2%	0.4%
I16	4.8%	2.0%	0.4%
I17	6.0%	<0.2%	<0.2%
I20	11.2%	<0.2%	<0.2%
I23	1.4%	<0.2%	<0.2%
I25	22.4%	<0.2%	<0.2%
I27	9.0%	<0.2%	<0.2%
I29	3.6%	<0.2%	<0.2%
I31	9.6%	<0.2%	<0.2%
I34	92.2%	>20.0%	<0.2%

CARBONATE SPECIMENS

Specimen No.	% Weight Loss	% Calcium	% Magnesium
I4	22.8%	3.0%	1.1%
I6	48.0%	6.0%	1.15%
I11	21.6%	3.0%	6.4%
I14	44.2%	7.0%	5.0%
I18	75.2%	14.0%	24.0%
I19	37.8%	5.0%	4.0%
I21	61.8%	8.4%	8.0%
I22	91.2%	16.0%	15.2%
I30	15.0%	1.9%	1.6%
I35	50.8%	6.6%	13.4%
I37	51.0%	4.4%	24.0%

TALC SPECIMENS

Specimen No.	% Weight Loss	% Calcium	% Magnesium
I2	3.6%	<0.2%	0.4%
I3	1.6%	<0.2%	<0.2%
I5	5.4%	<0.2%	<0.2%
I8	6.0%	<0.2%	<0.2%
I9	<0.2%	<0.2%	<0.2%
I10	4.2%	<0.2%	<0.2%
I24	8.0%	<0.2%	<0.2%
I26	<0.2%	<0.2%	<0.2%
I28	12.6%	<0.2%	<0.2%
I32	1.2%	<0.2%	0.4%
I33	5.6%	0.34%	<0.2%
I36	4.6%	<0.2%	<0.2%

/Continued....

J&J-0130588

TALC SPECIMENS (Continued)

Specimen No.	% Weight Loss	% Calcium	% Magnesium
I38	1.0%	<0.2%	<0.2%
I39	<0.2%	<0.2%	<0.2%
I40	7.0%	<0.2%	<0.2%
I41	<0.2%	<0.2%	<0.2%
I42	0.8%	<0.2%	<0.2%
I43	6.2%	<0.2%	<0.2%
I44	<0.2%	<0.2%	<0.2%
I45	8.0%	<0.2%	<0.2%

J&J-0130589

JNJAZ55_000017609

Electron Microscope Examination of Italian
Mine Samples and Imported Batch Shipments of
Italian Powder

The main purpose of the electron microscope examination of mine samples and also representative fractions of the Italian powder has been to establish whether or not any particles corresponding to the commercial forms of asbestos were present. The electron microscope is an instrument which is most usefully employed in the examination of particles less than ten microns in size. It has been used in this investigation therefore to examine only the finer particulate portion of the Italian samples. It may be argued that only a small fraction of each of the powdered samples was examined and that this was not representative of the total sample. However, we can assume that the fraction examined was representative of the dust formed from each sample and that it is this finer fraction which is the most important from a biological standpoint. Also as the size of the biologically active commercial asbestos particles fall entirely within the particle size range examined we can consider the main aim of the examination to be entirely satisfied by only looking at the finer fractions from each of the Italian samples.

To acquaint ourselves with the type of particles formed by the commercial asbestos minerals, Figs. A-D have been included. They represent samples of Amosite, Crocidolite, Anthophyllite and Chrysotile asbestos. Also Figs. E-F have been inserted to demonstrate typical single particle electron diffraction patterns which can be obtained from the four asbestos types for comparison with patterns obtained from the Italian samples.

Sample Preparation

Small portions of the powdered rock samples and imported powder specimens were placed in 15cc centrifuge tubes to which distilled water was added. The powders were then dispersed first by hand shaking and then with the aid of a small ultrasonic bath. The concentration of suspended material in the tubes was adjusted by eye using dilutions of distilled water. The tubes containing suspended solids were then allowed to stand for 20 minutes to allow the larger particles of mineral to sediment to the bottom of the tubes.

Electron microscope grids coated with carbon films were prepared and small drops of the particulate material from each of the specimen tubes were mounted on specimen grids and allowed to dry. The specimens were inserted into an A.E.I. E.M.6. electron microscope and examined for particles resembling commercial asbestos fibres. Where suitable particles were observed, selected area electron diffraction patterns were taken and compared with those produced by the commercial asbestos minerals. In all cases photomicrographs representative of the type of particles found in each sample were taken while interesting diffraction patterns were also recorded.

J&J-0130590

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Particle Morphology

The carbonate rich materials were found to produce compact particles which were very electron dense. On the whole they were finer particles than those obtained after crushing talc rich specimens. No fibrous material whatsoever was found when carbonate material only was comminuted. The morphology of particles produced from the footwall rocks i.e. limestone, marble, gneiss and the amphibolites were also very compact, although in the gneiss specimen platy particles were present probably representing the muscovite content of the specimen. Again in the footwall rock specimens fibrous particles were very scarce. Those lath like particles detected resembled the amphibole minerals rather than chrysotile. Selected area diffraction patterns which were obtained from the lath like particles in no way resembled the typical amphibole fibre diffraction pattern. They were generally very distorted patterns containing streaks rather than spots indicating a rather stressed and deformed material.

The specimens which were composed of talc together with other mineral associations, presented a very different picture, as far as particle shape was concerned. In the main particles were flat and plate-like, some being very thin and translucent in the electron beam. Particle sizes varied from very small to quite large plates some with very sharp discrete edges, others with rather ragged outlines. Comparing particles from those samples of talc which varied in bulk morphology in hand specimens, no observable difference could be drawn between them. Similarly, a comparison of particles produced from talc specimens of varying colour revealed no differences in the overall particle shape. Similarly those specimens rich in chlorite did not form particles with any distinctive features.

There were, however, observable differences in particle morphology between individual powder specimens. In the main most produced good plate like particles, however, one or two specimens were found to contain considerable numbers of lath like particles, these being very thin in character. These particles resembled the amphibole asbestos type particle being less regular and also very much larger in projected diameter. Diffraction patterns from these particles matched those obtained from the platy particles with which they were associated and in no way resembled the typical amphibole diffraction pattern obtained from single amphibole asbestos fibres.

Other fibrous particles were observed in the mainly talc specimens which to some extent resembled chrysotile asbestos fibres rather than amphibole minerals. They often had a somewhat textile appearance but were, however, crystalline. Diffraction patterns from these fibres were very distorted and in no way matched typical chrysotile or amphibole patterns.

The only group of specimens in which amphibole fibres were confirmed were in those specimens with known amphibole composition. However, even the fibres found in these specimens barely resembled the fibres formed by the commercial amphibole asbestos minerals. To assess the particles produced from the pure amphibole mineral (Tremolite), found in three of the specimens, small crystals of the mineral were taken from the hand specimens and crushed separately. An examination of the finer particles produced revealed stubby electron dense fibres associated with irregular lumps of the same mineral. Diffraction patterns from these fibres were similar to those obtained from the commercial amphibole minerals, although they were more difficult to obtain because of the greater thickness of these particles. Other specimens in the group, which did not contain talc but were composed of sheet silicate minerals mainly muscovite, were also practically free of fibrous particles. There appeared to be no general tendency for these other minerals to form fine fibrous particles. A number of very fine short fibres were observed on grids prepared from several of the talc specimens, these were, however, chance small pieces torn from the edges of talc plates. They appeared in those samples which had a tendency to form copious numbers of very fine particles when subjected to comminution.

The specimens examined can be grouped into four categories on the basis of particle morphology and they are as follows:

- (a) Talc specimens with impurities of carbonate and chlorite.
- (b) Rock type specimens, i.e. footwall limestone etc.
- (c) Those specimens composed mainly of carbonates.
- (d) Amphibole specimens with carbonate and talc.

The talc specimens were characterised by the large number of plate like particles often translucent in the electron beam. Rock specimens varied from specimens which were composed mainly of compact electron dense particles to those with some sheet silicate content in which plate like particles become apparent. Those specimens composed mainly of carbonate material produced compact rounded particles, often very small and grouped together in aggregates. Finally the specimens containing amphibole were characterised by the compact nature of the particles with evenly distributed fibres and very few translucent plates. The groups of particles described are illustrated by the following micrographs which illustrate the various forms.

Selected area electron diffraction patterns obtained from single particles of the amphibole mineral are also presented showing the similarity of these patterns to those obtained from commercial asbestos fibres. Also included are single crystals patterns and polycrystalline patterns, from talc, chlorite and muscovite rich specimens. It can be seen that they are very different in character to those obtained from the amphibole mineral. However, patterns from the sheet silicate minerals mentioned above are all very similar and it is impossible to identify each of these minerals from their

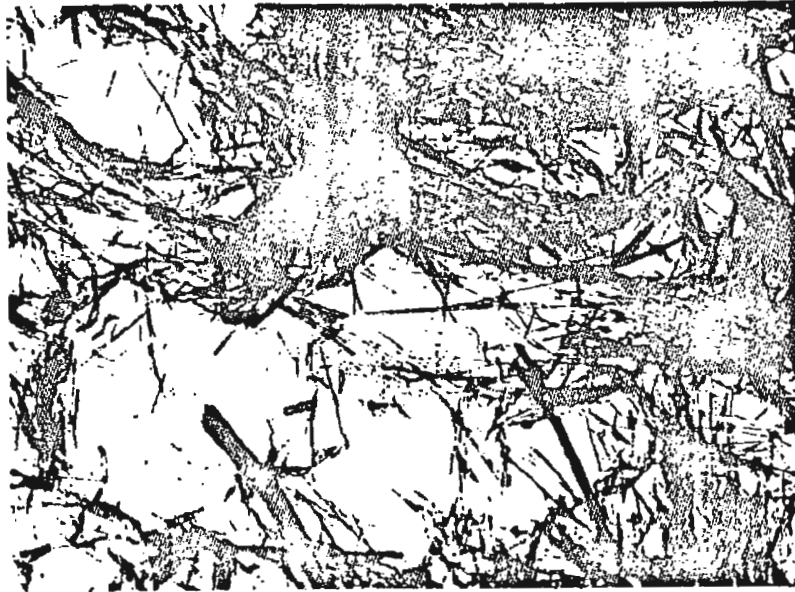
electron diffraction patterns or to tell them apart without applying a more sophisticated approach to the diffraction procedure. With specimen tilt facilities enabling the particle to be rotated through more than 45° discrimination is possible between certain of these minerals.

As mentioned earlier, patterns obtained from lath like particles found in the talc specimens were identical to those observed from general plate like forms. Those fibres with a textile like appearance often only gave very streaked patterns but in one or two cases these also resembled very closely the normal talc pattern.

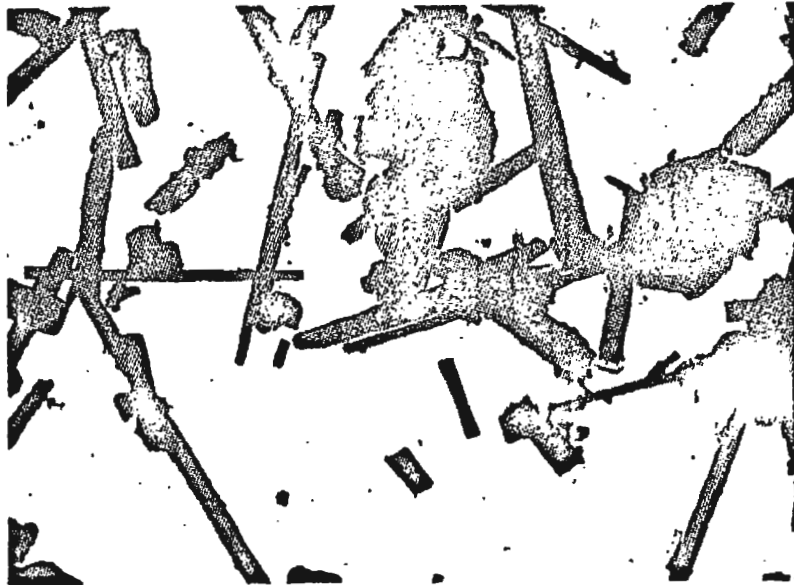
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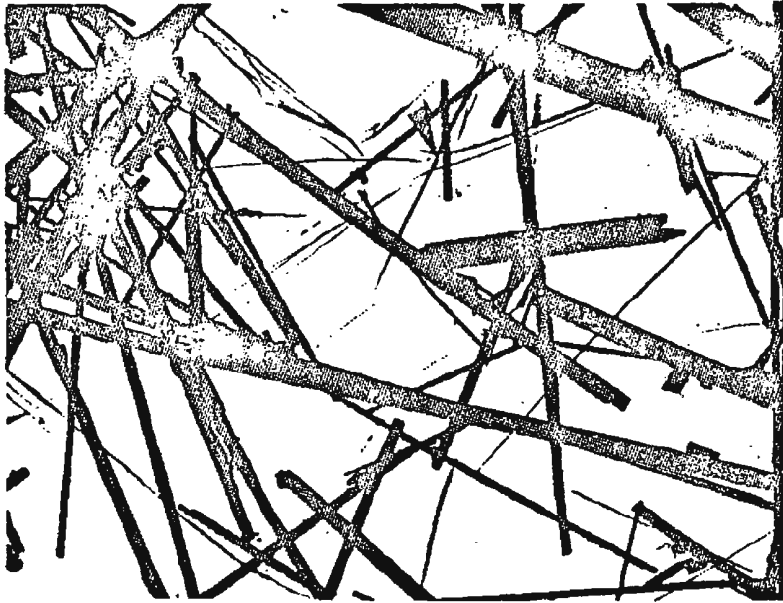
Examples of Commercial Amphibole and Chrysotile asbestos particles together with typical selected area electron diffraction patterns.



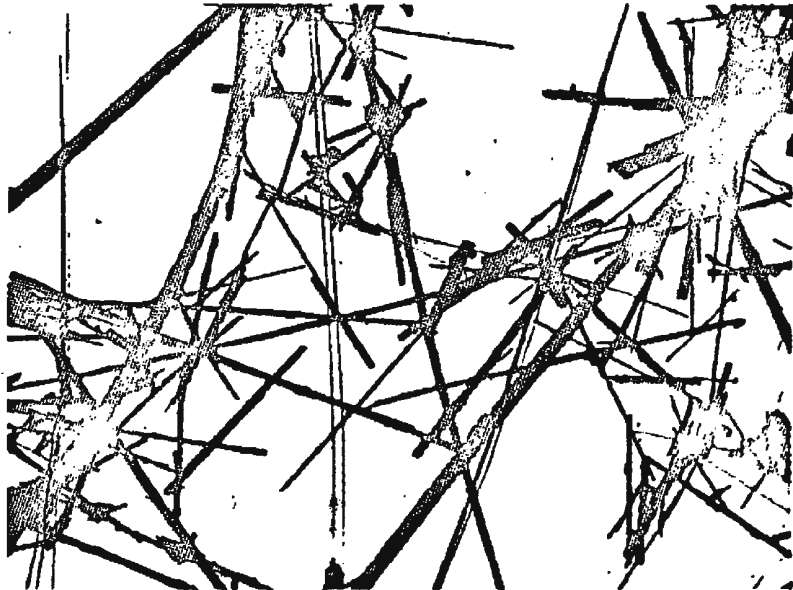
Chrysotile asbestos particles x 3000



Anthophyllite asbestos particles x 3000

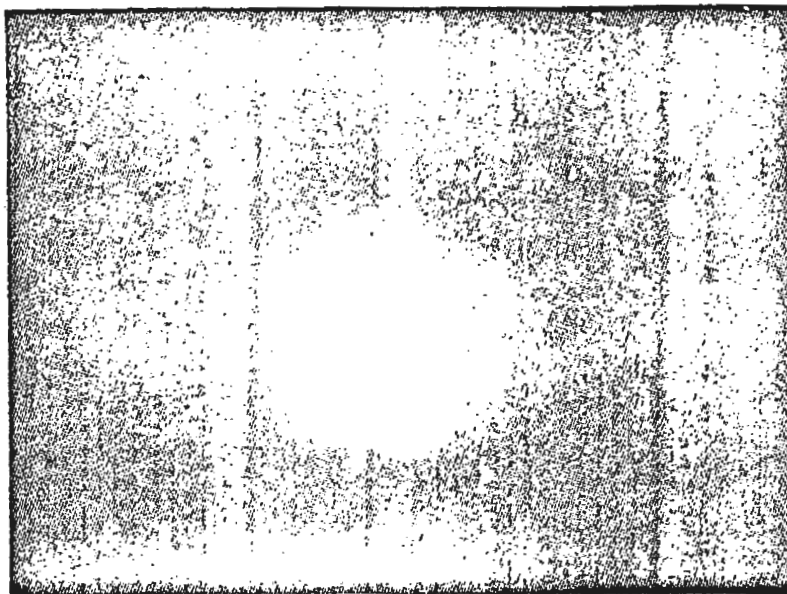


Amosite asbestos particles x 3000

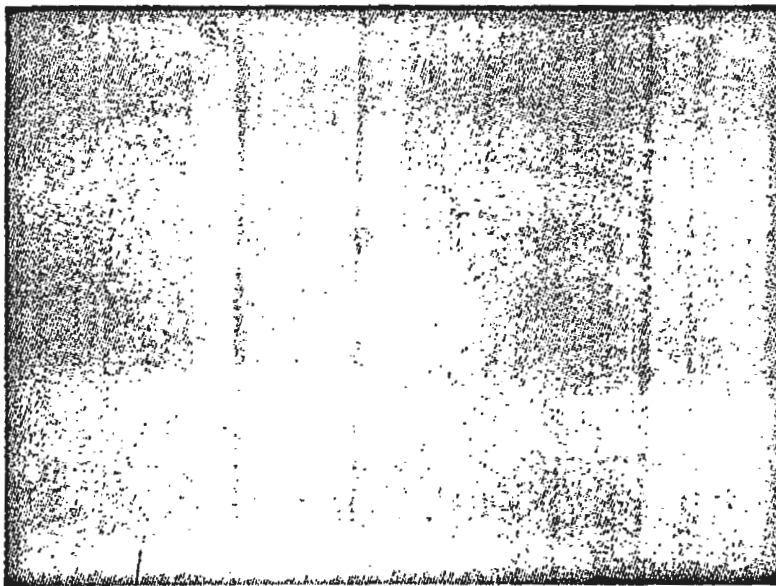


Crocidolite asbestos particles x 3000

J&J-0130595



Amphibole asbestos selected area
electron diffraction pattern.



Chrysotile asbestos selected area
electron diffraction pattern.

J&J-0130596

Electron micrographs of particles produced from specimens which have been classified as rock types.

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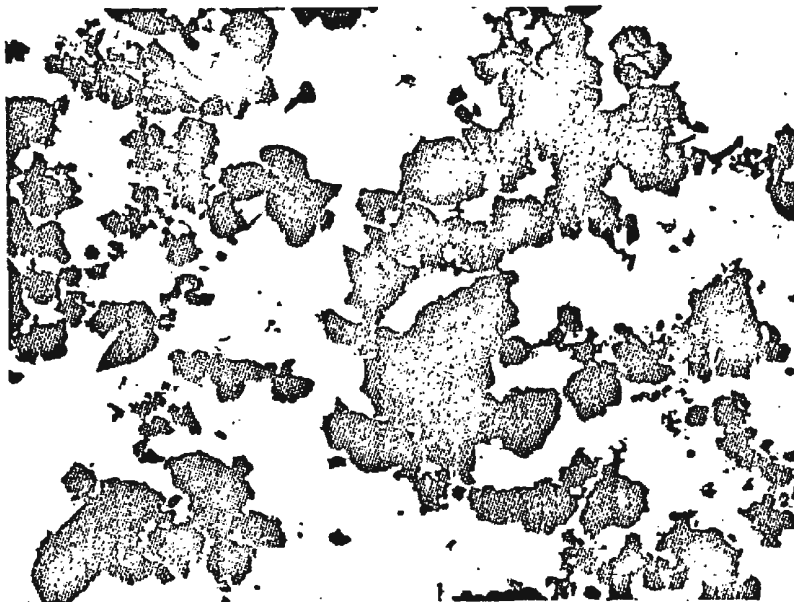


Fig. 1. Specimen I13 seam inclusion showing passage into talc x 3000. The particles are mainly compact and electron dense. A few flakes, no fibres present.

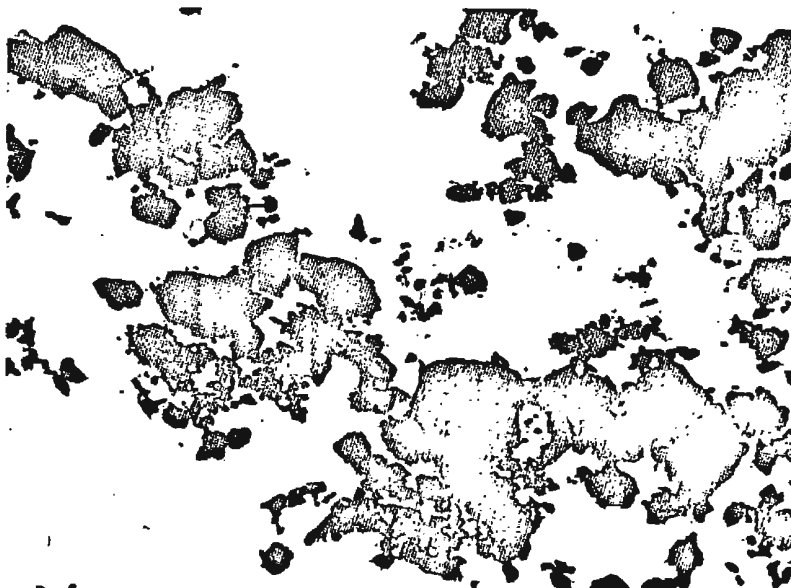


Fig. 2. Specimen I15. Talc footwall contact. x 3000. Compact particles with a few small flakes. No fibres present.

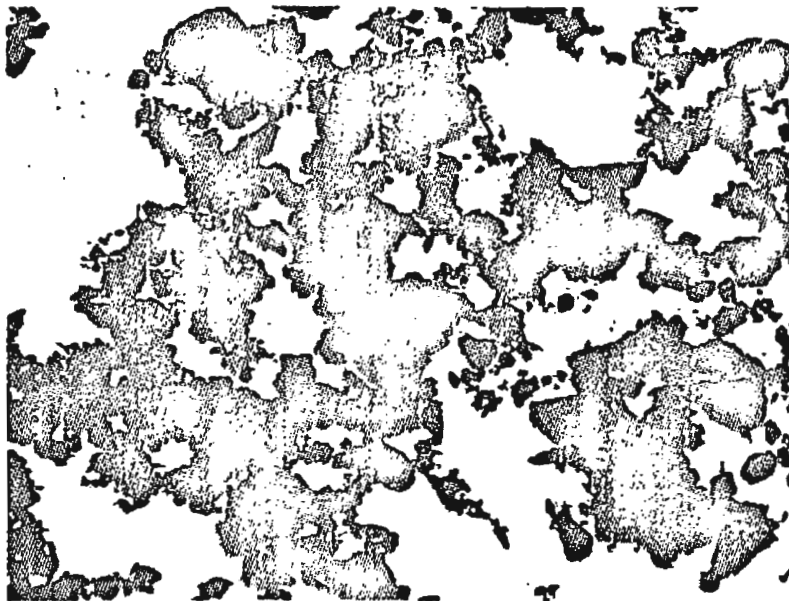


Fig. 3. Specimen I16. Lithological inclusion from Face 1. x 3000. Compact electron dense particles. No fibres present.

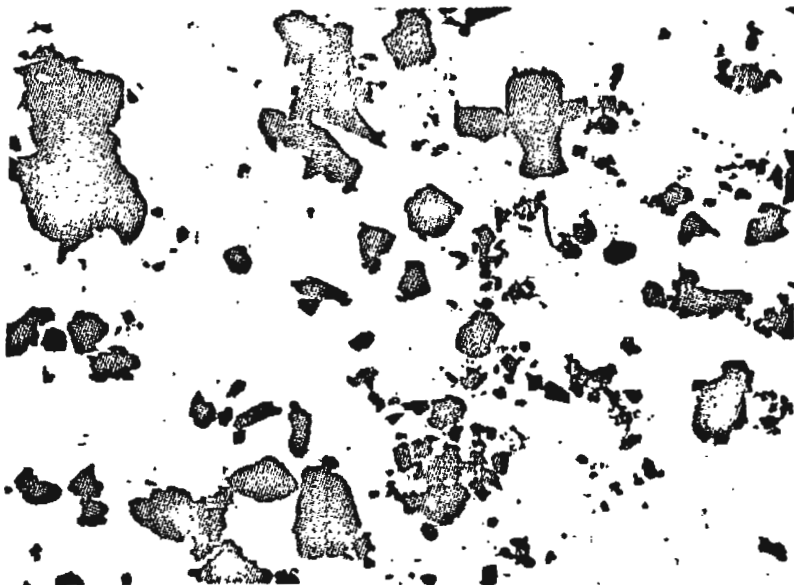


Fig. 4. Specimen I17. Footwall rock sample, x 3000. Mainly compact particles produced with a few plate like forms.

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Fig. 5. Specimen I23. Black gneiss, 2ft below talc seam. x 3000. Compact electron dense particles produced.

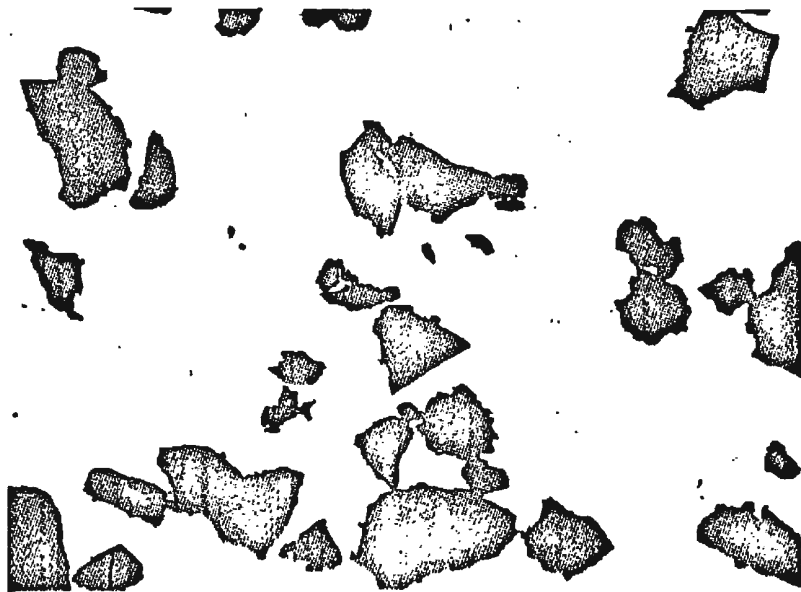


Fig. 6. Specimen I25. Footwall limestone. x 3000. Compact electron dense particles.

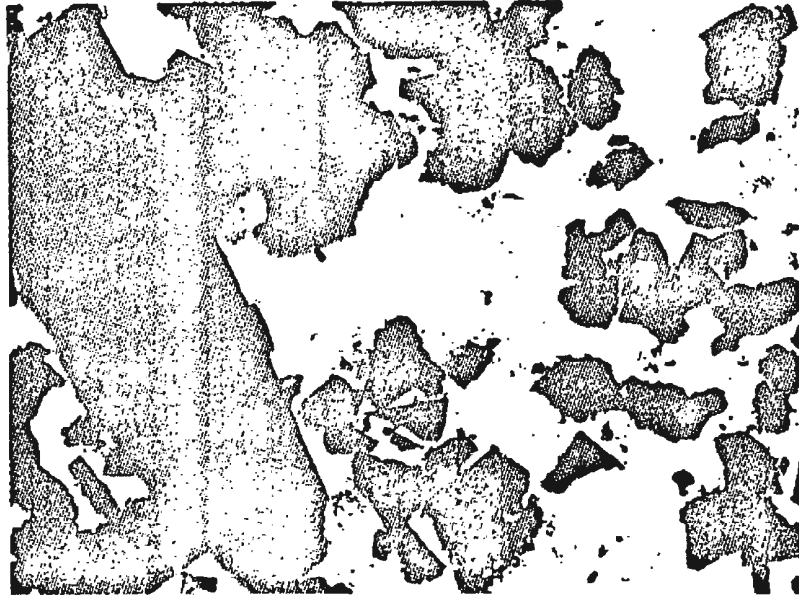


Fig. 7. Specimen I27. Lithological inclusion face 1.
x 3000. Platey electron dense particles.
No fibres.



Fig. 8. Specimen I29. Sample 6 Footwall. x 3000
Compact electron dense particles with a few
plate-like forms.

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Fig. 9. Specimen I31. Black inclusion face 1. x 3000
A mixture of plate-like and compact forms
mainly electron dense in character.

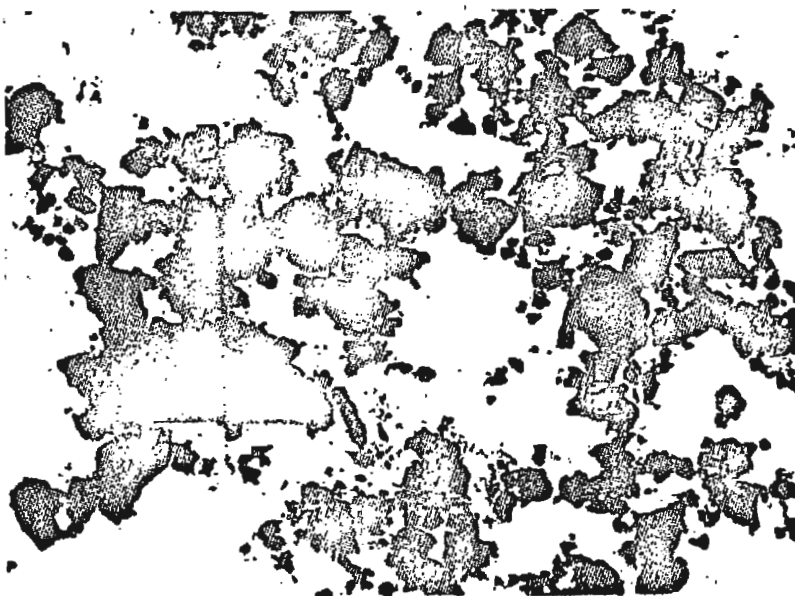


Fig.10. Specimen I34. Marble from tunnel wall. x 3000
Mainly compact electron dense particles with a
few plate-like forms.

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Electron micrographs of particles produced from those specimens mainly composed of carbonate minerals.



Fig. 1. Specimen I₁₁. Carbonate inclusion with some talc. x 3000. Particles consist of a mixture of compact and plate-like forms.

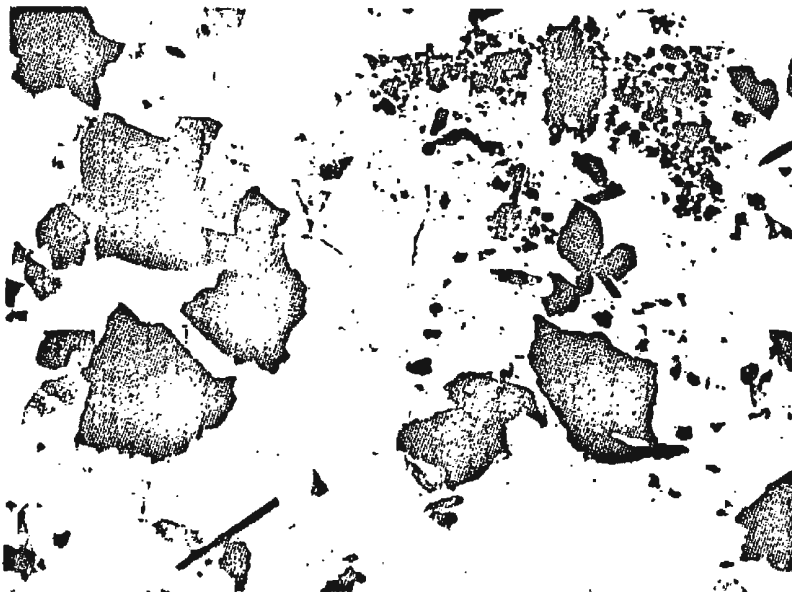


Fig. 2. Specimen I₁₄. Inclusion in talc seam Face 4, middle of seam. x 3000. Granular particles with plate-like types and lath-like forms.

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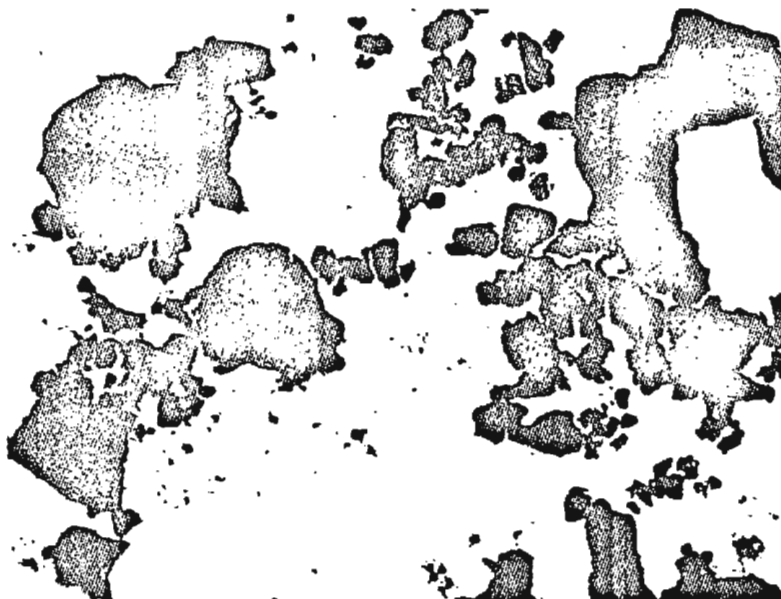


Fig. 3. Specimen I18. Carbonate/talc sample, x 3000. Particles compact and electron dense. A few plate-like forms.

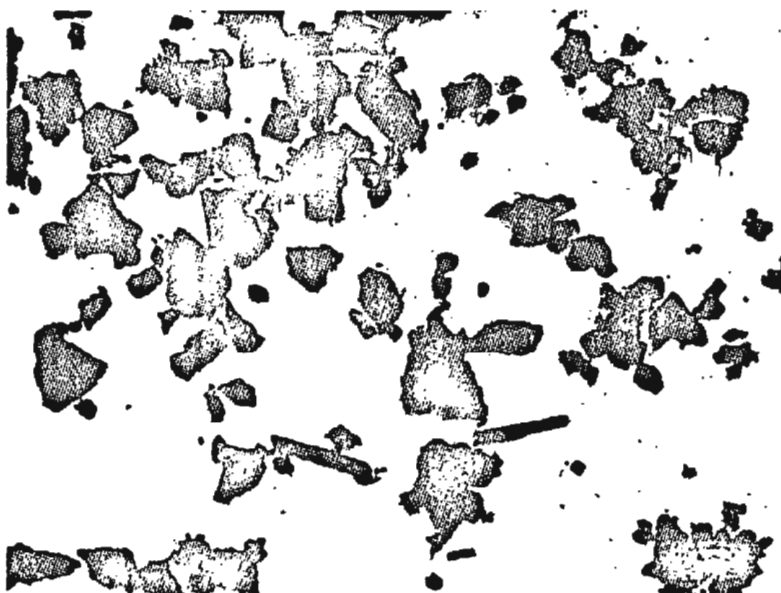


Fig. 4. Specimen I21. Inclusion from Face 2. x 3000. This specimen produced plate-like and compact particles with some lath-like forms.



Fig. 5. Specimen I35. Massive carbonate from rear end of working, x 3000. Compact electron dense particles with some plate-like talc particles.



Fig. 6. Specimen I37. Carbonate in talc inclusion x 3000. Compact particles together with some plate-like forms and rolled talc sheets.

Electron Micrographs of specimens of talc with
carbonate and other mineral inclusions.

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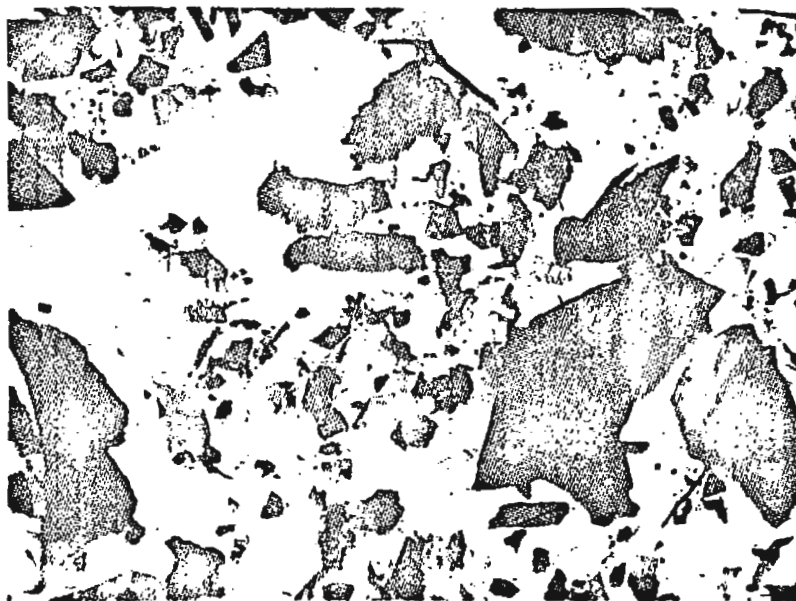


Fig. 1. Specimen I3. Coloured talc (Green) x 3000. Particles plate-like. Few fibres, rolled sheets and shords.



Fig. 2. Specimen I5. General ore, x 3000. Plate-like particles together with short lath-like particles, also a typical example of textile type fibre.

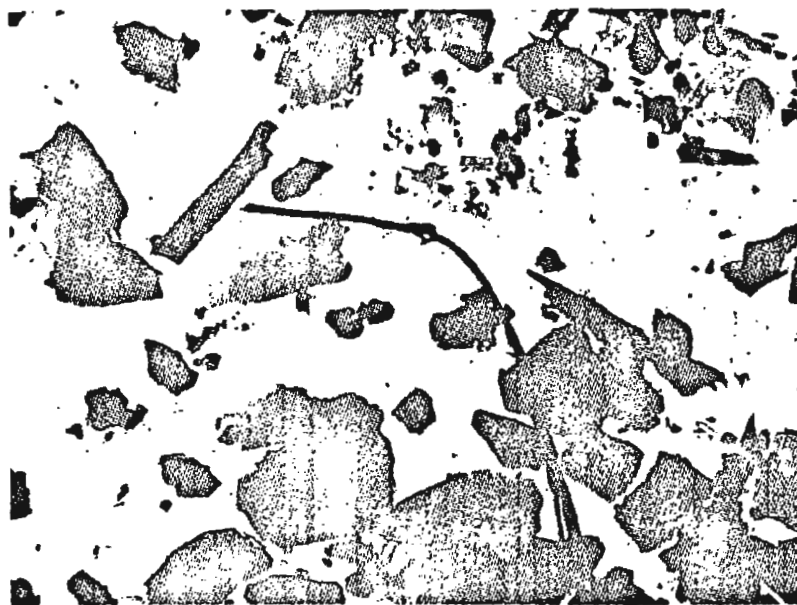


Fig. 3. Specimen Ig. Massive talc, x 3000. Plate-like particles with a few lath-forms also typical textile type long fibre.



Fig. 4. Specimen Ig. Grey talc First Face, x 3000. Practic ally all plate-like with a few lath forms.

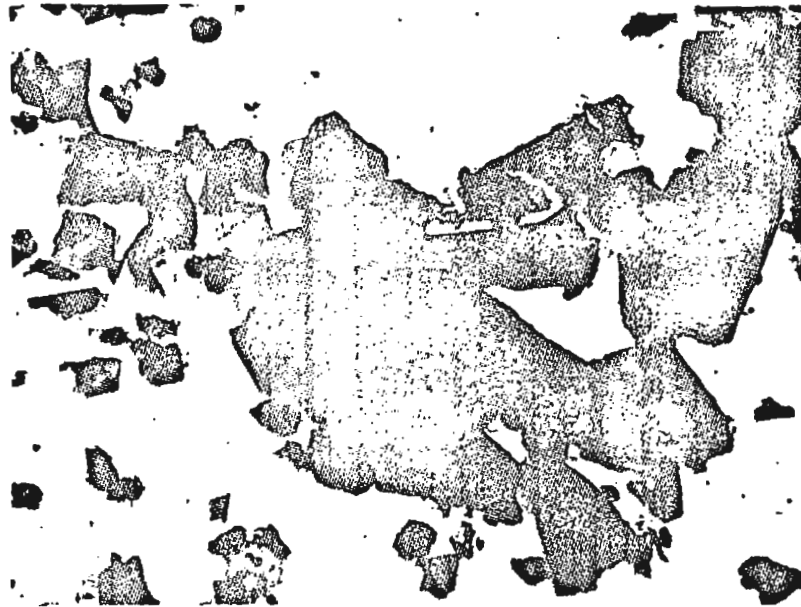


Fig. 5. Specimen I10. Granular talc, x 3000.
All plate-like particles.



Fig. 6. Specimen I24. Talc next to carbonate inclusion,
x 3000. This specimen was found to contain a
large number of lath-like particles, as can be
seen from the micrograph above. No diffraction
pattern corresponding with an amphibole fibre
was obtained from a selection of the elongated
particles.

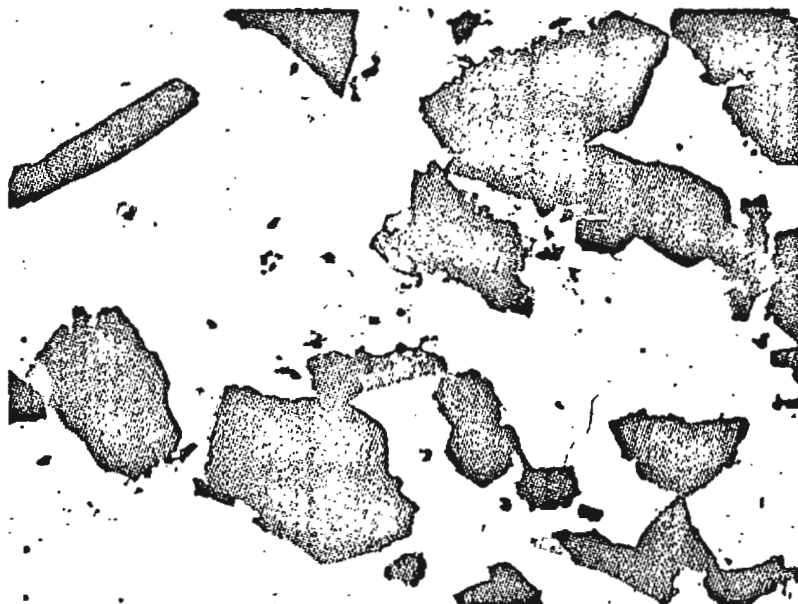


Fig. 7. Specimen I26. Coloured talc inclusions, x 3000. The particles produced from the various coloured inclusions in the talc were found to be mainly plate-like with a few lath forms.

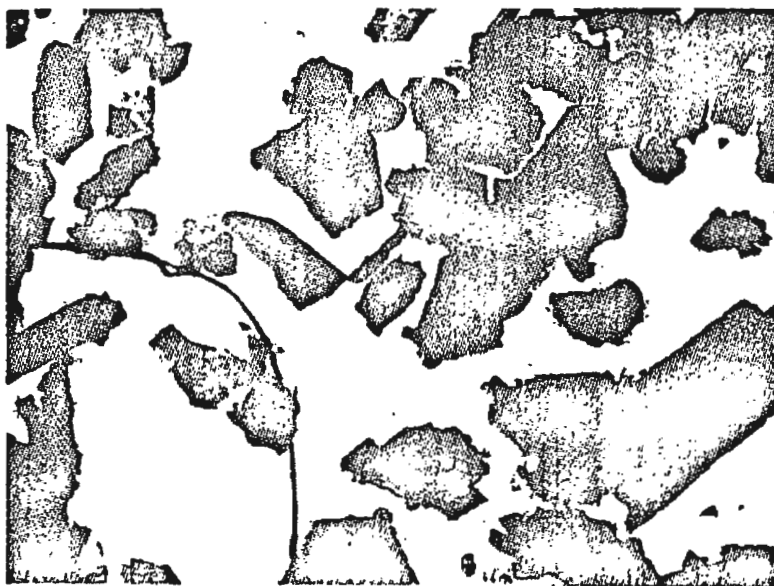


Fig. 8. Specimen I28. Talc/Quartz specimen, x 3000. Particles from this specimen were mainly plate-like but accompanied by more compact opaque particles. A few textile type fibres were observed.



Fig. 9. Specimen I32. Face 2 inclusion from base of talc seam, x 3000. The specimen produced a mixture of irregular particles varying from compact to plate-like in form with a few lath like particles.



Fig. 10. Specimen I33. Talc from lower left end of working x 3000. Particles mainly plate-like with some lath forms.

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Fig. 11. Specimen I38. Pyrite/Talc specimen, x 3000. Plate-like particles with some rolled tubes of talc.

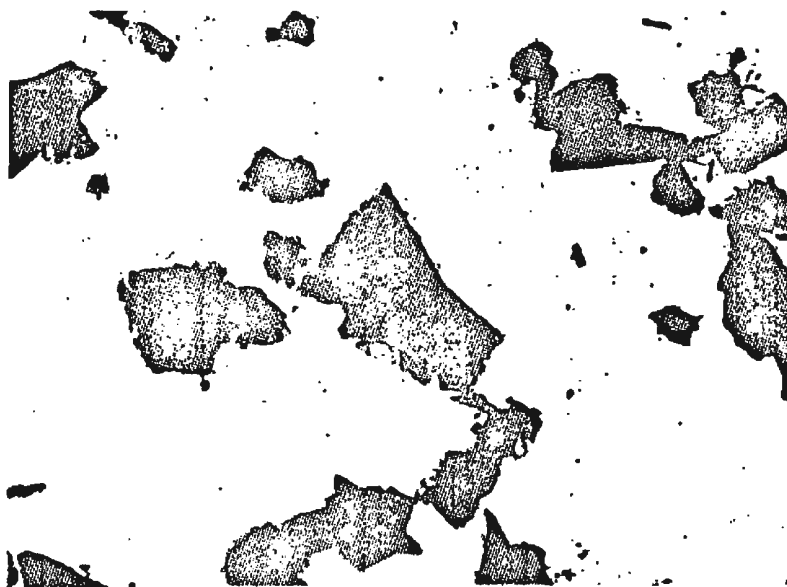


Fig. 12. Specimen I39. 5" - 0 coloured pieces from the crusher, x 3000. These various coloured talc pieces produced only plate-like particles.



Fig. 13 Specimen I41. Face 2, good talc specimen x 3000. Plate-like particles together with rolled talc sheets lath forms and textile type fibres.

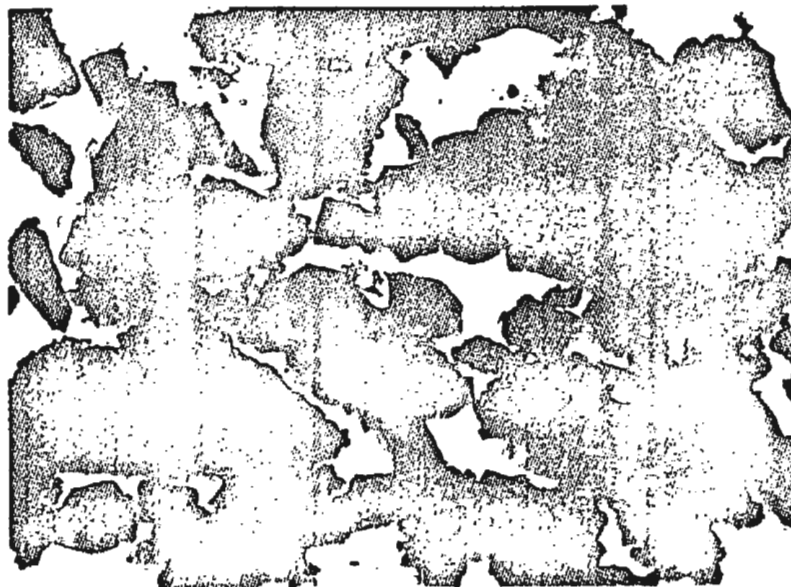


Fig. 14. Specimen I42. Face 1, green coloured talc, x 3000. This coloured specimen produced plate-like particles which were rather more electron dense.



Fig. 15. Specimen I43. Face 10. Fibrous looking hand specimen, x 3000. This sample was found to be practically all plate-like in form.



Fig. 16. Specimen I44. Face 1. Pure talc sample, x3000. Plate-like particles with some lath-like forms.

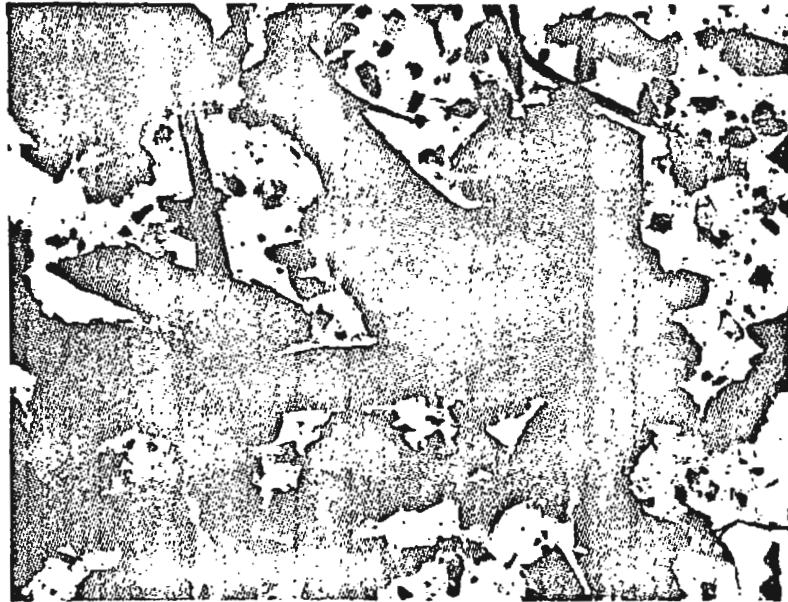


Fig. 17. Specimen I45. Face 1. Good talc specimen, x 3000. A mixture of plate-like particles and fibrous forms, including rolled tubes and textile type fibres.

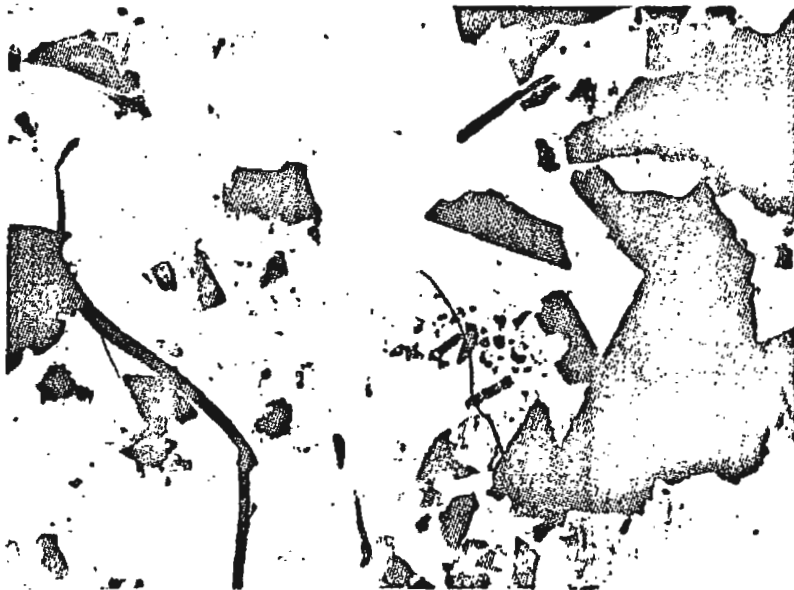


Fig. 18. Specimen I46. Face 3. Coloured specimen x 3000. Plate-like particles with shards and lath like forms, together with a typical textile form, which can be seen to have a sheet-like form.

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Electron Micrographs of particles produced from those specimens containing amphibole mineral and also from the amphibole mineral itself.

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Fig. 1. Specimen I19. Tremolite/carbonate talc sample x 3000. Compact particles, a few lath forms present.

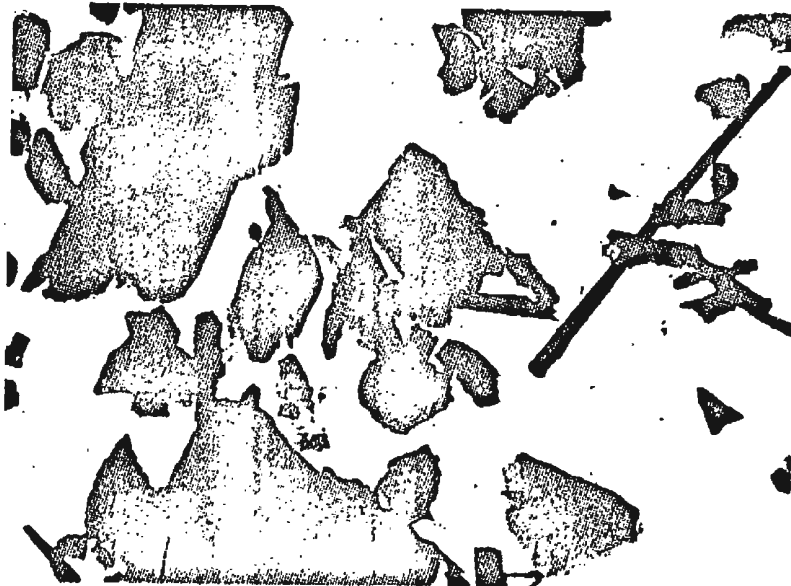
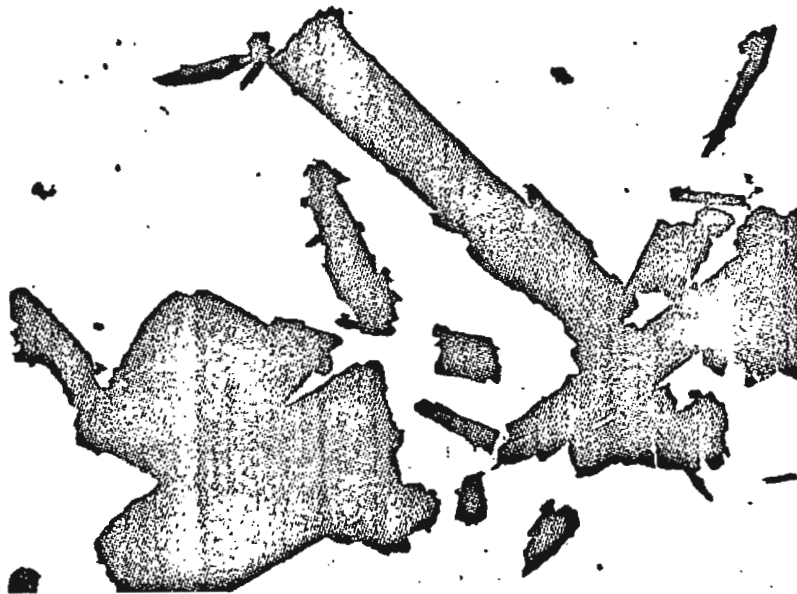


Fig. 2. Specimen I20. Amphibole sample from Guiana level 1212. x 3000. Compact particles with numerous lath forms.

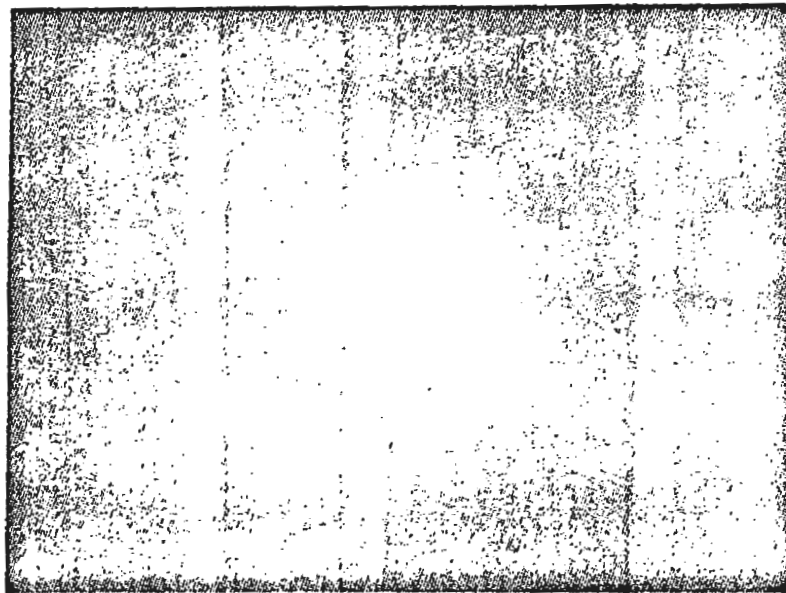
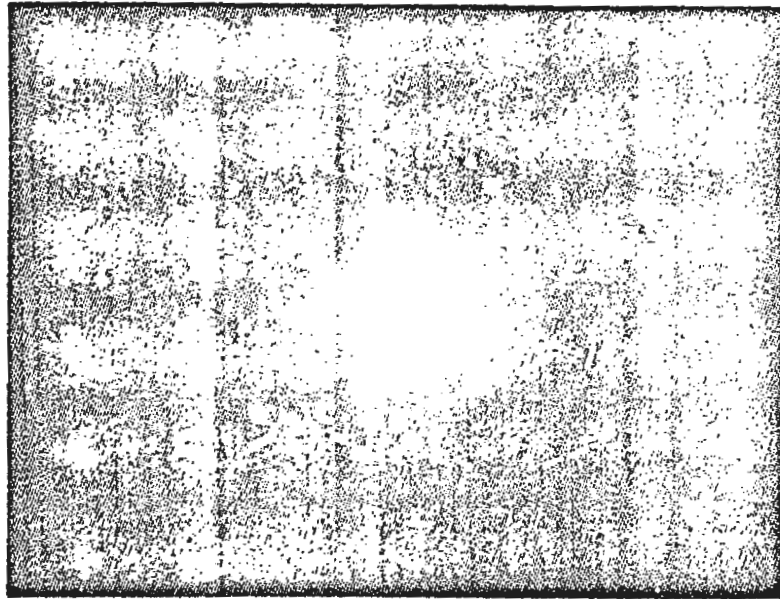


Figs. 3 and 4

Particles produced from single crystals of tremolite extracted from specimens I19 and I20. x 3000. Very few fibrous particles were produced when this specimen was crushed. Those that were fibrous in nature were thick and stubby in character, less than 50% of the particles were elongated in shape.

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Figs. 5 and 6

Selected area electron diffraction patterns
obtained from amphibole particles found in
specimens I19 and I20.

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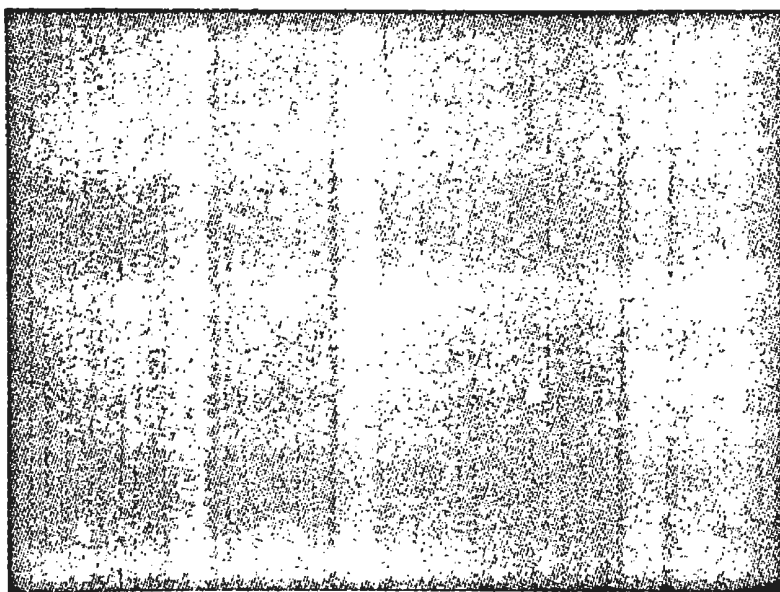


Fig. 7. Typical selected area diffraction pattern obtained from talc plates.

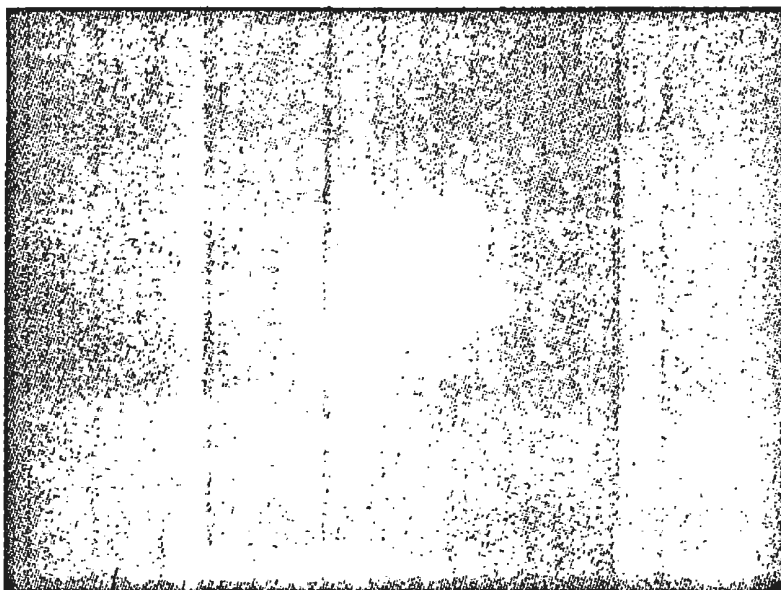


Fig. 8. Selected area diffraction pattern obtained from a typical textile type fibre showing features of a rotated or coiled structure.

X-RAY ANALYSIS OF ITALIAN MINE SAMPLES

Introduction

This report concerns the X-ray powder analysis of the Italian mine samples. The samples were classified into three categories according to their chemical and physical properties:

- (i) 'Rock' Type
- (ii) 'Talc' Type
- (iii) 'Carbonate' Type

All the samples were prepared by similar means and the procedure for obtaining the X-ray powder patterns was standardised.

From these powder photographs, several were chosen which clearly showed distinct mineral phases. These were used as standards for this group of samples. These standard patterns were compared against the ASTM index and this comparison illustrates the need to prepare standards for a particular locality from specimens at that locality.

The samples were compared with these standards by computer methods and visually and the results and discrepancies between the methods of comparison noted.

LIST OF SAMPLES

See Table 1

SAMPLE PREPARATION

The samples were received mainly as large rocks and were labelled according to their appearance and location in the mine.

With the larger samples a section was cut from the middle to be a representative sample, for the smaller samples as many pieces as possible were crushed to form the representative sample.

These samples were then roughly broken up and placed in a 'Tema' disc mill and ground for 5 mins. until all the sample was below approx. 100 mesh. These powders were stored in clean plastic bags. The samples, when required for X-ray analysis, were further ground (to less than 300 mesh) in a small agate ball mill and then sieved through a 350 mesh screen and stored in plastic bags.

The grinding mills and other apparatus used were thoroughly cleaned between samples and during the grinding care was taken to obtain a good representative sample.

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X-RAY ANALYSIS

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All the samples were analysed using a Debye-Scherrer camera mounted on a Raymax RX 3-D X-ray generator. A copper X-ray tube was used with nickel filters (0.02 mm thick) and the power rating of the tube set at 36 kV and 22mA.

The apparatus was carefully aligned and checked before mounting a sample. All the samples had the same exposure time of 8 hrs.

The samples were loaded into 0.5 mm diameter Lindemann glass tubes to be mounted in the Debye-Scherrer cameras. In the cameras Ilford Industrial 'G' X-ray film was used. The film was processed using Kodak DX-80 developer and Ilford Hypain fixer. The films were developed for 5 minutes using a 1:4 dilution for the developer and fixed for 2 minutes. The films were then washed in running water for 30 minutes and allowed to dry naturally. The X-ray films were then measured.

Using an illuminated screen and the line-spacings calculated, taking into account film shrinkage, from these line spacings the bragg angle and 'd' spacings can be calculated.

STANDARD PATTERNS

When all the samples X-ray photographs had been measured and the 'd' spacings calculated, they were visually inspected to find the film showing samples with pure mineral phases. These patterns were then taken as standards.

The samples were then broken up and the different mineral phases were sorted by hand to attempt to find a purer standard. These samples were then crushed in a similar way to the samples crushed beforehand. For X-ray analysis they were placed in 0.2 mm diameter tubes and given a 12 hr exposure. This method was used to give finer lines on the X-ray photograph and the larger exposure was to try and detect as many impurities as possible.

The 'd' spacings of the standards were compared with the A.S.T.M. index and also with themselves. They were compared with themselves to check that all the Talc and Chlorite standards matched each other and were similar in intensity.

Several standards were prepared containing the same mineral. This was because the 'd' spacings of the mineral varied slightly from sample to sample and especially with chlorite, depending on its composition the major reflections varied between 13.5% and 15.0%. This was mainly due to varying iron content and this can easily be seen on the X-ray films as it causes fluorescence with copper radiation and blackens the X-ray film generally.

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RESULTS

For the analysis of the results the samples have been divided into five sections:

- (i) standard patterns
- (ii) sample patterns (rock type)
- (iii) sample patterns (carbonate type)
- (iv) sample patterns (talc type)
- (v) batch sample patterns (includes old powders and shipments).

Two methods were used to find the mineral present in the sample. One method uses a computer program to detect the mineral.

In this method the bragg angles of samples were compared with the bragg angles of the standard and the number of lines fitted printed out. A print out was also obtained of all the standards which fitted a particular line to find all the possible minerals present and to see which lines were common to several standards.

As this procedure is quite long, the lines in the sample were first sorted into order of decreasing intensity and then the three most intense lines of the sample compared with the standards. If all three lines failed to match it was considered that that standard was not present and so the program deleted that standard from the comparison. At the end of the program the list of the standards was printed with the percentage of lines fitted to the sample noted.

The obvious disadvantage of this comparison was that the program could take no account of the relative intensities of the lines and so a visual method was used to find which was the major mineral phase. The computer program usually found the mineral phases present in the samples but could not place them in the correct order.

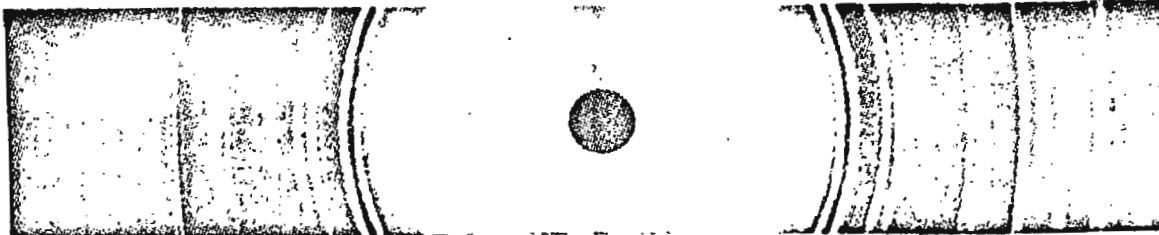
Patterns used as standards from the
Italian mine samples and their
comparison with A.S.T.M. data and
against themselves.

J&J-0130625

JNJAZ55_000017

SAMPLE SIP 1 TALC

544



Comparison against A.S.T.M. index: 1 line unmatched, 1.1145 Å

Patterns not included: 6-263 Muscovite -2M1, 7-25 Muscovite (1M), 7-32 Muscovite (2M1), 7-76 Ripidolite (Chlorite), 7-78 Thuringite (Chlorite), 7-166 Bavalite (Chlorite), 10-183 Peninnite Chlorite, 11-78 Dolomite, B and T Quartz.

Most probable minerals present: Talc Muscovite Calcite

Comparison against Italian Standards

Patterns not included: Chlorite (I42), Chlorite (I4), Muscovite (I35), Magnesite (I6), Tremolite (I19/I20), Dolomite.

Most probable minerals present: Talc

Visual comparison

Talc, Calcite

Minerals detected

Talc, Calcite

SAMPLE SIP 2 TALC



Comparison against A.S.T.M. index: 2 lines unmatched, 1.1159 Å
1.1353 Å

Patterns not included: 7-76 Ripodolite (Chlorite), 7-78 Thuringite (Chlorite), 7-166 Bavalite (Chlorite).

Most probable minerals present: Talc, Muscovite, Calcite

Comparison against Italian Standards

Patterns not included: Chlorite (I42), Chlorite (I4), Tremolite (I19/I20).

Most probable minerals present: Talc, Muscovite, Magnesite.

Visual Comparison

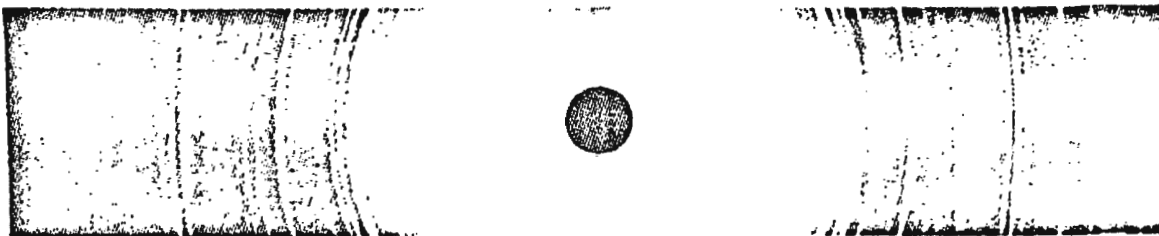
Talc, Chlorite, Magnesite

Minerals Detected

Talc, Chlorite, Magneitie

J&J-0130626

JNJAZ55_00001764



Comparison against A.S.T.M. index: 2 lines unmatched, 1.1739 $\text{\AA}$,
1.29 $\text{\AA}$

Patterns not included: 6-263 Muscovite -2M1, 7-25 Muscovite (IM)
7-32 Muscovite (2M1), 7-79 Forsterite (Olivine), 8-479 Magnesite

Most probable minerals present: Chlorite, Talc

Comparison against Italian Standards

Patterns not included: Muscovite (I35), Tremolite (I19 and I20)

Most probable minerals present: Chlorite, Talc.

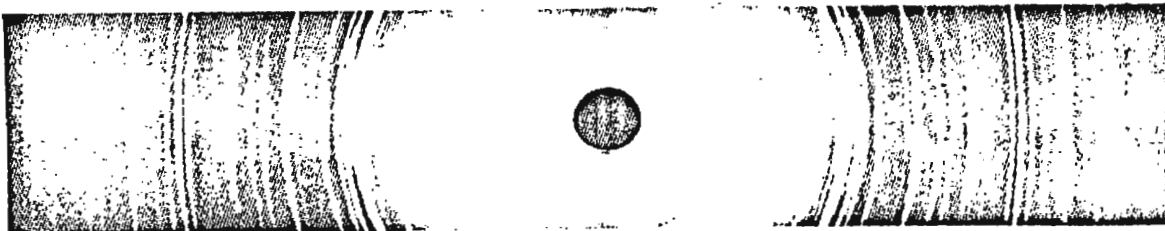
Visual Comparison

Chlorite, Talc

Minerals Present

Chlorite, Talc

SAMPLE SIP 4 CHLORITE



Comparison against A.S.T.M. index: 3 lines unmatched
1.1741 $\text{\AA}$, 1.1318 $\text{\AA}$, 1.0984 $\text{\AA}$.

Patterns not included: 6-263 Muscovite -2M1, 7-32 Muscovite
(2M1), 8-479 Magnesite, 11-78 Dolomite, 13-437 Boric Acid.

Most probable minerals present: Chlorite, Talc

Comparison against Italian Standards

Patterns not included: Calcite (I34), Magnesite (I37),
Muscovite (I35), Tremolite (I19/I20), Dolomite.

Most probable mineral present: Chlorite, Talc

Visual Comparison

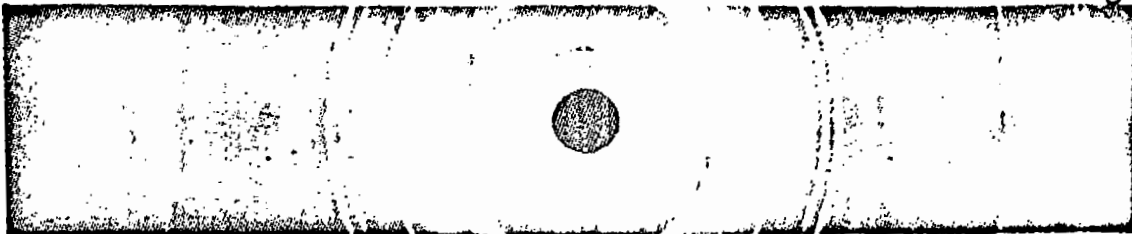
Chlorite, Talc

Minerals Present

Chlorite, Talc

SAMPLE SIP 5 TALC

546



Comparison against A.S.T.M. index:

Patterns not included: 5-586 Calcite, 7-25 Muscovite (IM),
7-77 Sheridanite (Chlorite), 7-79 Forsterite (Olivine),
7-166 Bavalite (Chlorite).

Most probable minerals present: Talc, Muscovite, Chlorite

Comparison against Italian Standards

Patterns not included: Chlorite (I42), Chlorite (I4),
Magnesite (I6), Tremolite (I19/I20).

Most probable minerals present: Talc

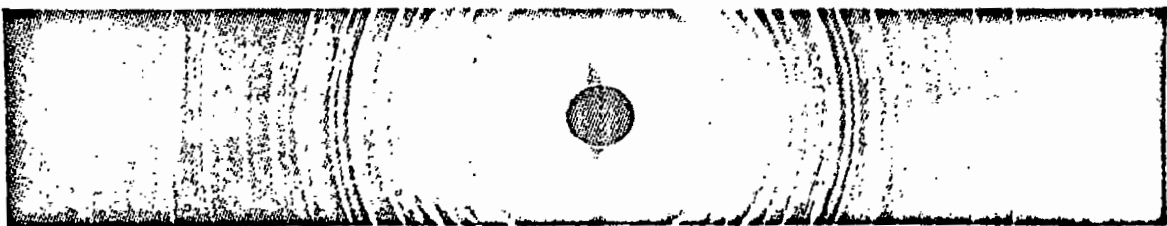
Visual comparison

Talc, Chlorite

Minerals Present

Talc, Chlorite

SAMPLE SIP 6 MUSCOVITE



Comparison against A.S.T.M. index: 3 lines unmatched, 1.7999^o_A,
1.3721^o_A, 1.2741^o_A.

Patterns not included: 3-881 Talc, 7-79 Forsterite (Olivine),
7-166 Bavalite (Chlorite), 7-183 Penninite (Chlorite),
8-479 Magnesite, 11-78 Dolomite, 19-770 Talc.

Most probable minerals present: Muscovite, Chlorite

Comparison against Italian Standards

Patterns not included: Magnesite (I37), Tremolite (I19 and I20),
Dolomite

Most probable minerals present: Muscovite, Talc

Visual Comparison

Muscovite, Calcite

Mineral Present

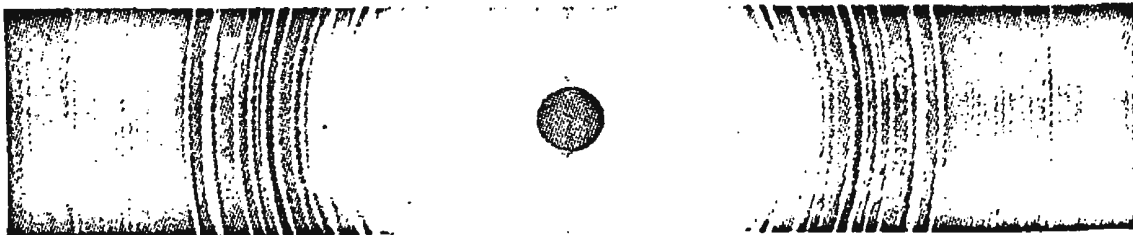
Muscovite, Calcite

J&J-0130628

JNJAZ55_00001764

SAMPLE SIP 7 MAGNESITE

547



Comparison against A.S.T.M. Index: 1 line unmatched 1.1092⁰_A

Patterns not included: 5-586 Calcite, 6-263 Muscovite -2M1, 7-25 Muscovite (IM), 7-32 Muscovite (2M1), 7-160 Chlorite (Kotshubeite), 7-76 Ripodolite (Chlorite), 7-78 Thuringite (Chlorite), 7-166 Bavalite (Chlorite), 10-183 Penninite Chlorite, 13-437 Tremolite.

Most probable minerals present: Magnesite, Dolomite, Talc

Comparison against Italian Standards

Patterns not included: Calcite (I34), Chlorite (I4)
Muscovite (I35), Tremolite (I19/I20).

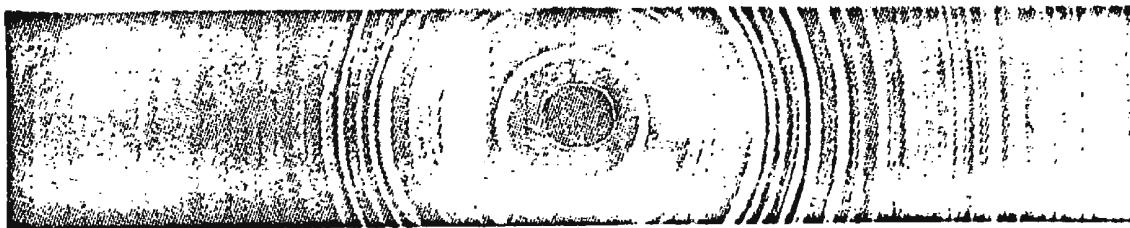
Most probable minerals present: Magnesite, Dolomite, Talc

Visual Comparison

Magnesite, Talc

Minerals Present

Talc, Magnesite.

SAMPLE SIP 8 TREMOLITE

Comparison against A.S.T.M. Index: 1 line unmatched 1.1118⁰_A

Patterns not included: 6-263 Muscovite -2M1, 7-25 Muscovite (IM), 7-32 Muscovite (2M1), 7-42 Muscovite (3T), 7-79 Forsterite (Olivine).

Most probable minerals present: Tremolite, Talc, Calcite

Comparison against Italian Standards

Patterns not included: Magnesite (I37), Chlorite (I4),
Muscovite (I35).

Most probable minerals present: Tremolite, Talc, Calcite

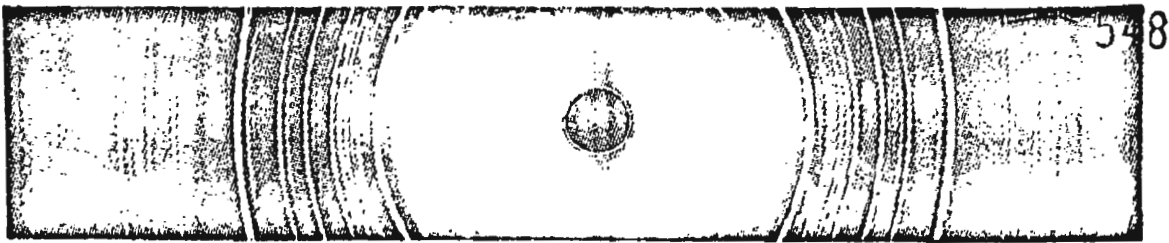
Visual Comparison

Tremolite, Talc

Minerals Present

Tremolite, Talc

22
SAMPLE SIP 9 DOLOMITE



Comparison against A.S.T.M. Index: 1 line unmatched 1.1094Å⁰

Patterns not included: 3-881 Talc, 6-263 Muscovite -2M1, 7-25 Muscovite (IM), 7-32 Muscovite (2M1), 19-814 Muscovite 2M1 (Vanadian), 7-160 Chlorite (Kotschubeite), 7-79 Forsterite (Olivine), 13-437 Tremolite, 19-770 Talc.

Most probable minerals present: Dolomite, Muscovite

Comparison against Italian Standards

Patterns not included: Magnesite (I37), Chlorite (I4) Tremolite (I19/I20).

Most probable minerals present: Dolomite, Talc

Visual Comparison

Dolomite, Muscovite, Calcite

Minerals Present

Dolomite, Muscovite, Calcite

SAMPLE SIP 10 CALCITE



Comparison against A.S.T.M. Index: 3 unmatched lines
1.2095Å⁰, 1.1098Å⁰, 1.0926Å⁰

Patterns not included: 7-160 Chlorite (Kotschubeite), 7-79 Forsterite (Olivine), 13-437 Tremolite.

Most probable minerals present: Calcite, Muscovite

Comparison against Italian Standards

Patterns not included: Magnesite (I6), Tremolite (I19-I20).

Most probable minerals present: Calcite, Muscovite

Visual Comparison

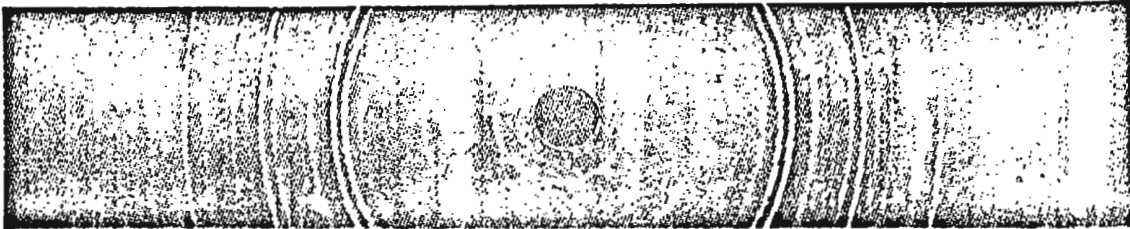
Calcite

Minerals Present

Calcite, Muscovite

J&J-0130630

JNJAZ55_000017650

SAMPLE SIP 11 MAGNESITE

Comparison against A.S.T.M. Index: 1 unmatched line 1.1085A⁰

Patterns not included: 5-586 Calcite, 7-25 Muscovite (IM),
7-160 Chlorite (Kotschubeite), 7-76 Ripidolite (Chlorite),
7-78 Thuringite (Chlorite), 7-166 Bavalite (Chlorite),
10-183 Penninite Chlorite, B & T Quartz.

Most probable minerals present: Magnesite, Dolomite, Talc

Comparison against the Italian Standards

Patterns not included: Calcite (I34), Chlorite (I4),
Muscovite (I35).

Most probable minerals present; Magnesite, Dolomite, Talc

Visual Comparison

Magnesite, Dolomite, Talc

Minerals Present

Magnesite, Talc, Dolomite

J&J-0130631

JNJAZ55_000017651

Examples of Patterns Obtained from
Rock Type Specimens and Their
Major Mineral Content from X-Ray
Comparison.

J&J-0130632

SAMPLE I1 TALC FROM FOOTWALL CONTACT

551

Comparison

Patterns not included: Magnesite (I37), Tremolite (I19/I20).

Most probable minerals present: Chlorite, Muscovite, Talc,
Dolomite.

Visual Comparison: Talc Chlorite, Calcite

Minerals Present: Talc Chlorite, Calcite.

SAMPLE I7 MICA SCHIST

Comparison

Patterns not included: Magnesite (I37), Talc (I46),
Tremolite (I19/I20).

Most probable minerals present: Muscovite, Talc, Quartz

Visual Comparison: Muscovite, Talc, Quartz

Minerals Present:

SAMPLE I12 FOOTWALL SAMPLE? AMPHIBOLITE

Comparison: 3 lines unmatched. $6.4653\text{\AA}$ $1.2819\text{\AA}$ $1.225\text{\AA}$

Patterns not included: Calcite (I34), Magnesite (I37),
Talc (I46), Talc (I5), Tremolite
(I19/I20).

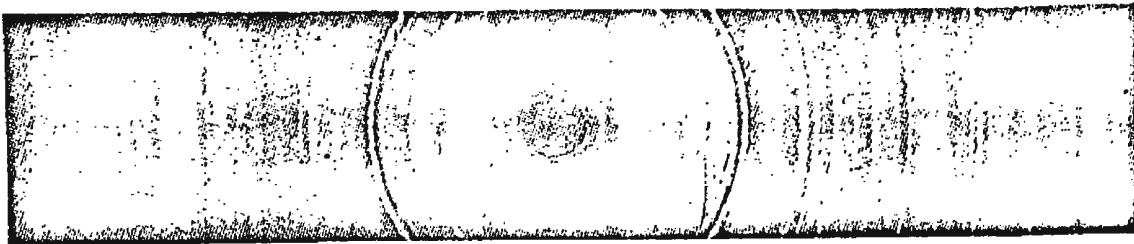
Most probable minerals present: Muscovite, Dolomite, Quartz.

Visual Comparison: Muscovite, Chlorite, Quartz

Minerals Present:

J&J-0130633

SAMPLE I13 INCLUSION SHOWING PASSAGE INTO TALC BOTTOM TRANSIT



552

Comparison: 1 unmatched line 1.1541 $\overset{\circ}{\text{A}}$

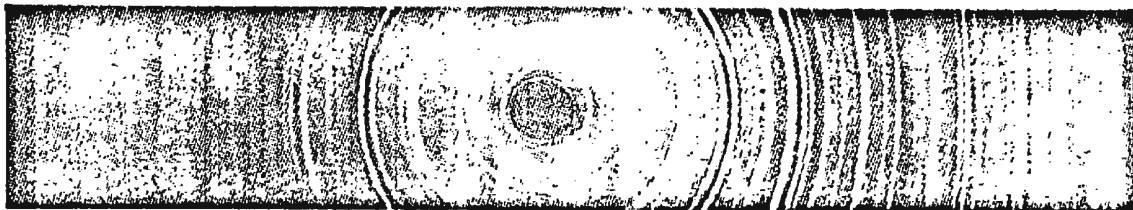
Patterns not included: Magnesite (I37), Muscovite (I35),
Tremolite (I19/I20), Dolomite

Most probable minerals present: Chlorite, Talc, Quartz

Visual Comparison: Chlorite, Muscovite, Quartz

Minerals Present: Chlorite, Muscovite, quartz

SAMPLE I15 TALC-FOOTWALL CONTACT



Comparison:

Patterns not included: Magnesite (I37), Tremolite (I19/I20).

Most probable minerals present: Chlorite, Talc, Muscovite,
Quartz.

Visual Comparison: Chlorite, Talc, Quartz

Minerals Present: Chlorite, Talc, Quartz

J&J-0130634

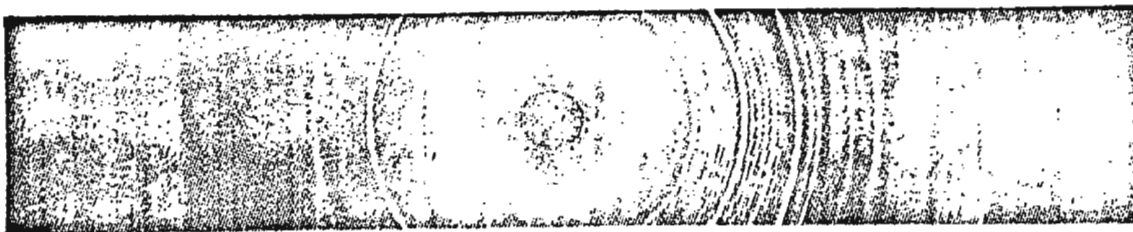
SAMPLE I16 FACE 1 INCLUSION BELOW SEAMComparison

Patterns not included: Talc (I45), Tremolite (I19/I20)
Dolomite

Most probable minerals present: Muscovite, Chlorite,
Calcite, Quartz

Visual Comparison: Chlorite, Muscovite, Calcite, Quartz

Minerals Present: Chlorite, Muscovite, Calcite, Quartz

SAMPLE I17 FOOTWALL ROCK SAMPLE

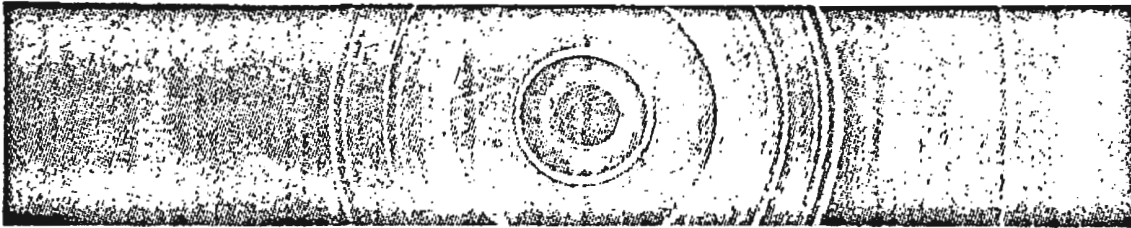
Comparison: 2 unmatched lines $6.6957\text{\AA}$, $1.6305\text{\AA}$

Patterns not included: Talc (I46), Chlorite (I42),
Muscovite (I35), Magnesite (I6), Tremolite (I19/I20), Dolomite.

Most probable minerals present: Calcite, Talc, Quartz

Visual Comparison: Calcite, Talc, Quartz

Minerals Present: Calcite, Talc, Quartz

SAMPLE I20 AMPHIBOLE SAMPLE FROM GUIANA LEVEL 1212

Comparison: 1 unmatched line 1.6309Å⁰

Patterns not included: Chlorite (I42), Chlorite (I4),
Muscovite (I35), Magnesite (I6), Dolomite.

Most probable minerals present: Talc, Tremolite, Calcite,
Magnesite.

Visual Comparison: Talc, Tremolite, Chlorite

Minerals Present: Talc, Chlorite, Tremolite

SAMPLE I23 BLACK GNEISS

Comparison: 5 unmatched lines 6.3586Å⁰, 1.449Å⁰, 1.2278Å⁰,
1.2121Å⁰, 1.1520Å⁰.

Patterns not included: Calcite (I34), Tremolite (I19/I20)

Most probable minerals present: Muscovite, Talc, Magnesite,
Quartz

Visual Comparison: Muscovite, Magnesite, Quartz

Minerals Present: Muscovite, Magnesite, Quartz

SAMPLE I25 LIMESTONE FOOTWALL

555

ComparisonPatterns not included: Calcite (I34), Tremolite (I19/I20).Most probable minerals present: Talc, Chlorite, QuartzVisual Comparison: Talc, Magnesite, QuartzMinerals Present: Talc, Magnesite, QuartzSAMPLE I27 LITHOLOGICAL INCLUSIONComparisonPatterns not included: Chlorite (I42), Chlorite (I4),
Tremolite (I19/I20), Magnesite (I6),
DolomiteMost probable minerals present: Talc, Calcite, QuartzVisual Comparison: Talc, Calcite, QuartzMinerals Present: Talc, Calcite, QuartzSAMPLE I29 SAMPLE 6 FOOTWALLComparison: 2 unmatched lines 1.1526^oA, 6.3031^oAPatterns not included: Calcite (I34), Magnesite (I37),
Chlorite (I4), Talc (I5).Most probable minerals present: Muscovite, Quartz, Dolomite,
TalcVisual Comparison: Muscovite, QuartzMinerals Present: Muscovite, Quartz

J&J-0130637

JNJAZ55_000017657

SAMPLE I31 BLACK INCLUSION

Comparison: 1 unmatched line 1.2145A⁰

Patterns not included: Magnesite (I37), Talc (I5), Dolomite

Most probable minerals present: Muscovite, Calcite, Talc

Visual Comparison: Muscovite, Calcite

Minerals Present: Muscovite, Calcite

SAMPLE I34 TUNNEL WALL - MARBLE

Comparison

Patterns not included: Tremolite (I19/I20), Magnesite (I6)

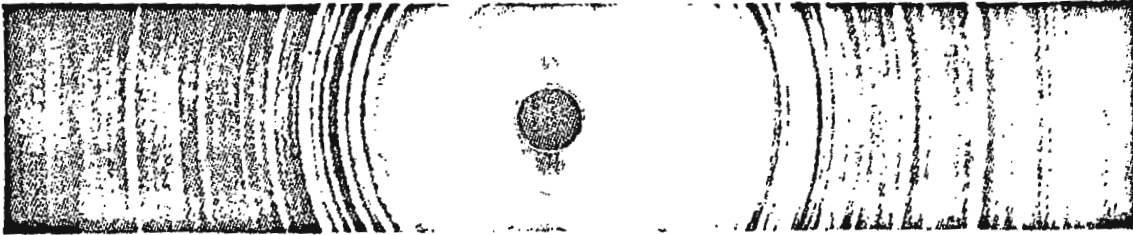
Most probable minerals present: Calcite, Muscovite, Talc

Visual Comparison, Calcite

Minerals Present Calcite

J&J-0130638

Examples of Patterns Obtained
from the Carbonate Specimens
and their Major Mineral Compo-
sition Obtained from Comparison
with Standards.

SAMPLE I4 FACE 10 AMPHIBOLE

Comparison: 3 unmatched lines $1.2586\overset{\circ}{\text{A}}$, $1.0823\overset{\circ}{\text{A}}$, $1.074\overset{\circ}{\text{A}}$

Patterns not included: Chlorite (I42), Chlorite (I4)
Dolomite

Most probable minerals present: Tremolite, Talc, Magnesite

Visual Comparison: Talc, Tremolite, Magnesite

Minerals Present: Talc, Tremolite, Magnesite

SAMPLE I6 QUARTZ

Comparison

Patterns not included: Calcite (I34), Chlorite (I4)
Tremolite (I19/I20)

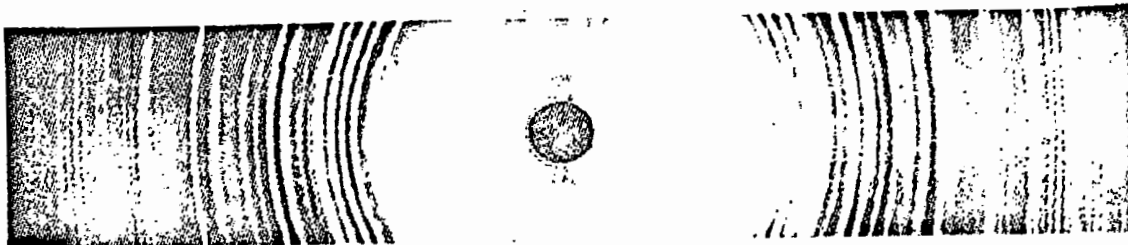
Most probable minerals present: Magnesite, Dolomite,
Talc

Visual Comparison: MAGNESITE, Talc

Minerals Present: Magnesite, Talc

J&J-0130640

JNJAZ55_000017

SAMPLE I11 CARBONATE - TALC INCLUSION

Comparison: 1 unmatched line 1.2143Å⁰

Patterns not included: Chlorite (I42), Chlorite (I4)

Most probable minerals present: Magnesite, Dolomite, Talc

Visual Comparison: Talc, Magnesite, Calcite

Minerals Present: Talc, Magnesite, Calcite

SAMPLE I14 SEAM 4 INCLUSION IN TALC

Comparison

Patterns not included: Magnesite (I37), Chlorite (I4),
Muscovite (I35), Tremolite (I19/I20)

Most probable minerals present: Dolomite, Talc

Visual Comparison: Talc, Dolomite

Minerals Present: Talc, Dolomite

SAMPLE I18 FACE 3 MAGNESITE AND TALC

Comparison:

Patterns not included: Talc (I5), Tremolite (I19/I20)

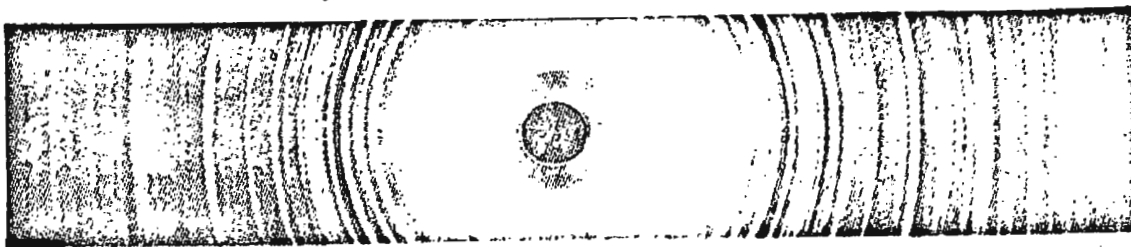
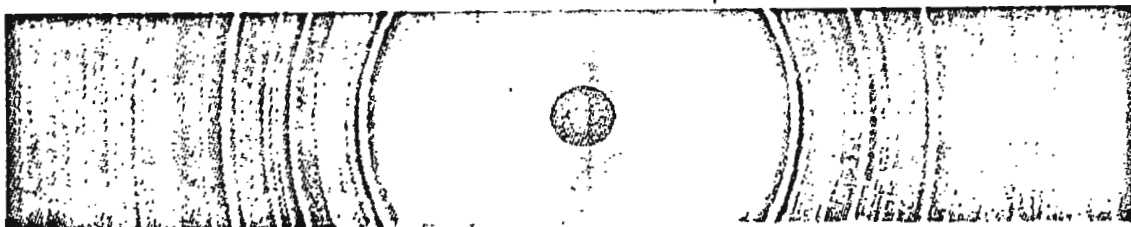
Most probable minerals present: Dolomite, Magnesite,
Chlorite

Visual Comparison: Dolomite, Talc Chlorite

Minerals Present: Dolomite, Talc, Chlorite.

J&J-0130641

JNJAZ55_000017661

SAMPLE I 19 IMPURITY IN TALC AND QUARTZComparison:Patterns not included: Magnesite (I37)Most probable minerals present: Tremolite, Dolomite,
Muscovite, TalcVisual Comparison: Talc, Tremolite, Magnesite.Minerals Present: Talc, Tremolite, MagnesiteSAMPLE I21 FACE 2 OCCLUSION (MAGNESITE)Comparison:Patterns not included: Calcite (I34), Chlorite (I4),
Muscovite (I35), Tremolite (I19/I20)Most probable minerals present: Dolomite, Magnesite, TalcVisual Comparison: Talc, Magnesite, DolomiteMinerals Present: Talc, Magnesite, Dolomite

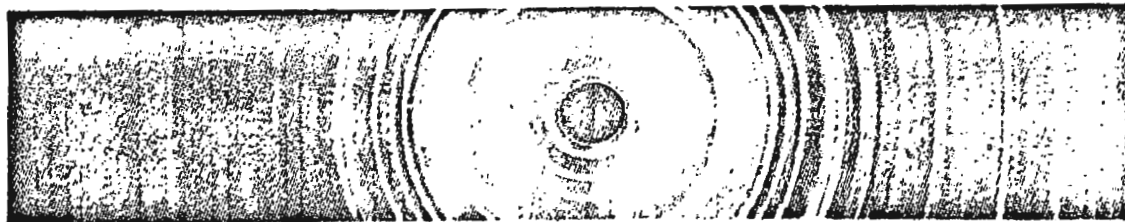
SAMPLE I22 MAGNESITE, DOLOMITE, TALCComparison:

Patterns not included: Calcite (I34), Talc (I45), Talc (I46)
Muscovite (I35), Tremolite (I19/I20).

Most probable minerals present: Dolomite, Magnesite,
Chlorite, Talc.

Visual Comparison: Talc, Dolomite.

Minerals Present: Talc, Dolomite

SAMPLE I30 TALC AND OTHERSComparison:

Patterns not included: Magnesite (I37), Talc (I5),
Tremolite (I19/I20).

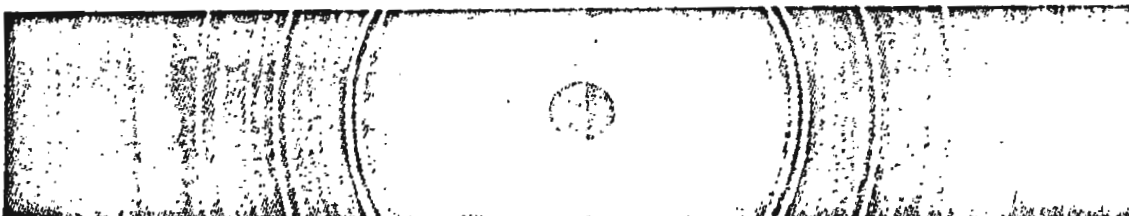
Most probable minerals present: Dolomite, Chlorite,
Muscovite, Talc.

Visual Comparison: Talc, Chlorite

Minerals Present: Talc, Chlorite

J&J-0130643

JNJAZ55_000017663

SAMPLE I35 MASSIVE CARBONATE. END OF WORKINGComparison:Patterns not included: Tremolite (I19/I20)Most probable minerals present: Muscovite, Magnesite,
ChloriteVisual Comparison: Magnesite, Talc, ChloriteMinerals Present: Magnesite, Talc, ChloriteSAMPLE I37 CARBONATE AND TALCComparisonPatterns not included: Calcite (I34), Chlorite (I4),
Muscovite (I35).Most probable minerals present: Magnesite, Dolomite, TalcVisual Comparison: Magnesite, TalcMinerals Present: Magnesite, Talc

J&J-0130644

JNJAZ55_00001766

Examples of Patterns and
Major Mineral Content of
Those Specimens Classified
as Talc Types Obtained by
Comparison.

J&J-0130645

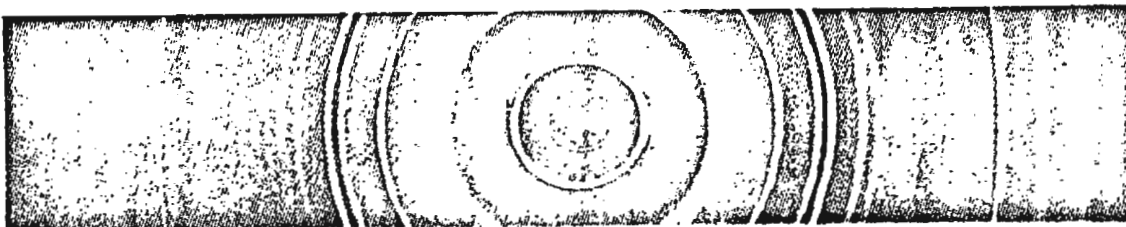
SAMPLE I2 SORTING PIECESComparison

Patterns not included: Tremolite (I19/I20).

Most probable minerals present: Chlorite, Magnesite, Talc

Visual Comparison: Chlorite, Talc

Minerals Present: Chlorite, Talc

SAMPLE I3 COLOURED TALCComparison:

Patterns not included: Chlorite (I42), Chlorite (I4),
Muscovite (I35), Magnesite (I6), Tremolite (I19/I20), Dolomite.

Most probable minerals present: Talc

Visual Comparison: Talc

Minerals present: Talc

SAMPLE I5 GENERAL ORE

Comparison: 2 unmatched lines 18.1157Å⁰ 7.0073Å⁰

Patterns not included: Chlorite (I42), Chlorite (I4),
Muscovite (I35), Dolomite.

Most probable minerals present: Talc, Magnesite

Visual Comparison: Talc

Minerals present: Talc

SAMPLE I8 MASSIVE TALC

Comparison

Patterns not included: Magnesite (I6), Tremolite (I19/I20).

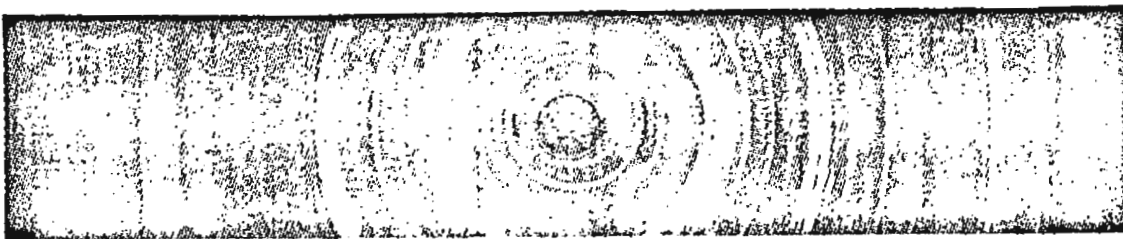
Most probable minerals present: Talc, Chlorite

Visual Comparison: Talc, Chlorite

Minerals Present: Talc, Chlorite

J&J-0130647

JNJAZ55_00001766

SAMPLE I9 FACE 1 GREY TALCComparison

Patterns not included: Calcite (I34), Magnesite (I37),
Muscovite (I35), Magnesite (I6), Tremolite (I19/I20).

Most probable minerals present: Talc, Chlorite

Visible Comparison: Talc, Chlorite

Minerals Present: Talc, Chlorite

SAMPLE I10 GRANULAR TALCComparison

Patterns not included: Calcite (I34), Magnesite (I37),
Chlorite (I42) Chlorite (I4),
Muscovite (I35), Magnesite (I6)
Tremolite (I19/I20)

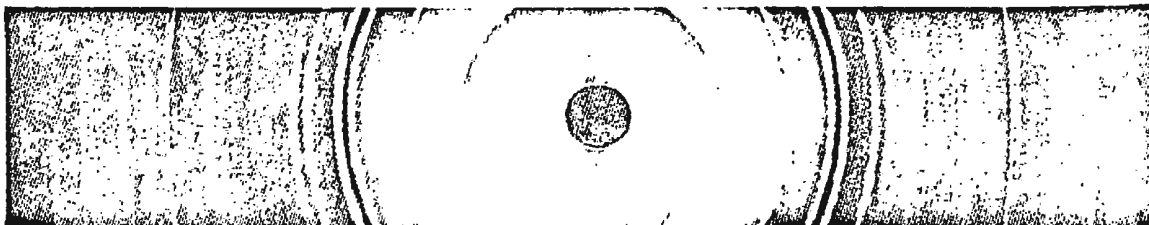
Most probable minerals present: Talc, Dolomite

Visible Comparison: Talc, Dolomite

Minerals Present: Talc, Dolomite

J&J-0130648

JNJAZ55_000017668

SAMPLE I24 TALC FACE 2Comparison:

Patterns not included: Muscovite (I35), Tremolite (I19/I20)
Magnesite (I6).

Most probable minerals present: Talc, Chlorite, Dolomite,
Magnesite

Visual Comparison: Dolomite, Magnesite, Talc

Minerals Present: Dolomite, Magnesite, Talc

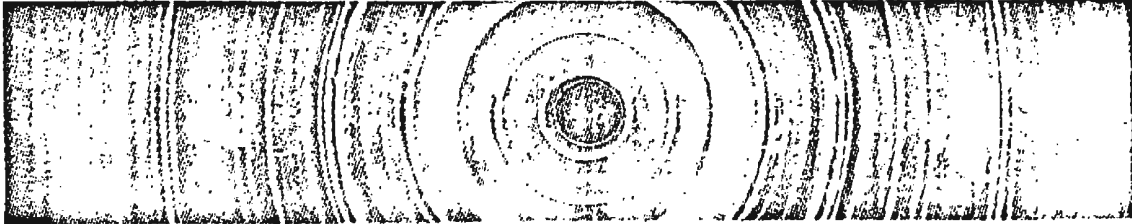
SAMPLE I26 TALC INCLUSIONSComparison

Patterns not included: Calcite (I34), Tremolite (I19/I20)

Most probable minerals present: Talc, Chlorite, Dolomite

Visual Comparison: Talc, Chlorite

Minerals Present: Talc, Chlorite

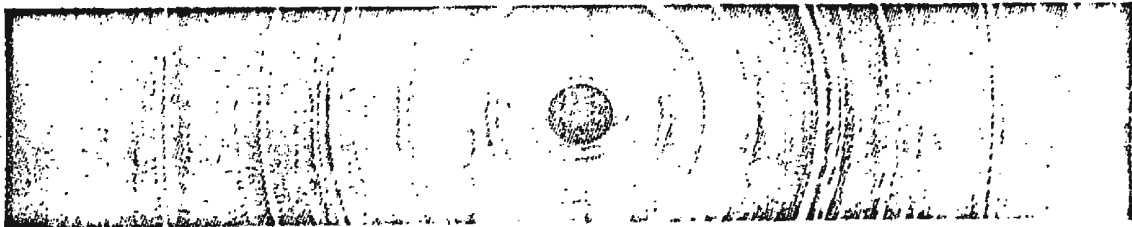
SAMPLE I28 QUARTZ TALCComparison

Patterns not included: Muscovite (I35), Tremolite (I19/I20)
Magnesite (I6), Dolomite

Most probable minerals present: Chlorite, Talc, Quartz

Visual Comparison: Chlorite, Talc, Quartz

Minerals Present: Chlorite, Talc, Quartz

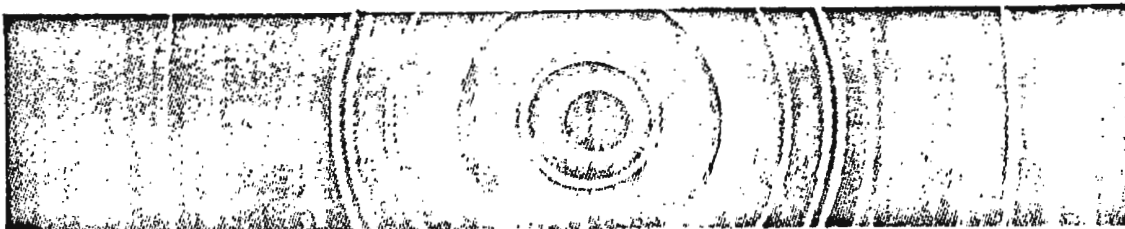
SAMPLE I32 OCCLUSION FACE 2Comparison

Patterns not included: Muscovite (I35), Tremolite (I19/I20)
Dolomite

Most probable minerals present: Chlorite, Talc, Magnesite

Visual Comparison: Chlorite, Talc

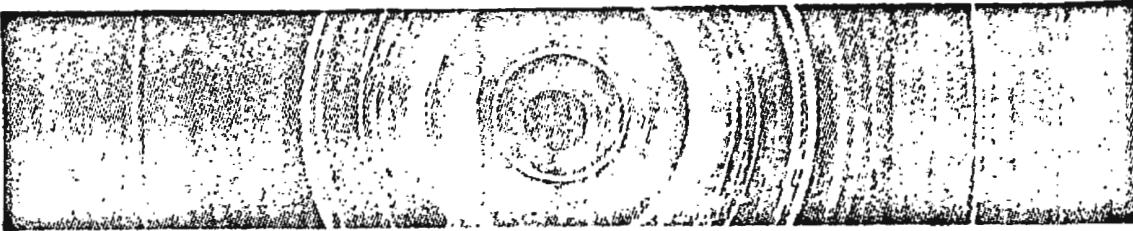
Minerals Present: Chlorite, Talc

SAMPLE I33 TALC END OF WORKINGComparison:Patterns not included: Muscovite (I35), Tremolite (I19/I20)Most probable minerals present: Talc, Chlorite, Magnesite
DolomiteVisual Comparison: Talc, Chlorite, MagnesiteMinerals Present: Talc, Chlorite, MagnesiteSAMPLE I36 GREY TALCComparison: 2 unmatched lines $1.2204\text{\AA}$, $1.1517\text{\AA}$ Patterns not included: Calcite (I34), Talc (I46)
Tremolite (I19/I20).Most probable minerals present: Chlorite, Muscovite, TalcVisual Comparison: Chlorite, TalcMinerals Present: Chlorite, TalcSAMPLE I38 TALC AND PYRITEComparison: 1 unmatched line $1.041\text{\AA}$ Patterns not included: Chlorite (I42), Chlorite (I4),
Muscovite (I35), Tremolite (I19/I20)Most probable minerals present: Talc, CalciteVisual Comparison: Talc, CalciteMinerals Present: Talc, Calcite

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SAMPLE I39 S-'Q' FROM CRUSHER

570

Comparison

Patterns not included: Muscovite, (I35), Tremolite (I19/I20),
Magnesite (I6).

Most probable minerals present: Talc Chlorite

Visual Comparison: Talc, Chlorite, Calcite

Minerals Present: Talc, Chlorite, Calcite

SAMPLE I40 PLATEY TALCComparison:

Patterns not included: Tremolite (I19/I20)

Most probable minerals present: Talc, Magnesite, Chlorite

Visual Comparison: Talc, Chlorite, Magnesite

Minerals Present: Talc, Chlorite, Magnesite

SAMPLE I41 GOOD SPECIMEN No.2.Comparison:

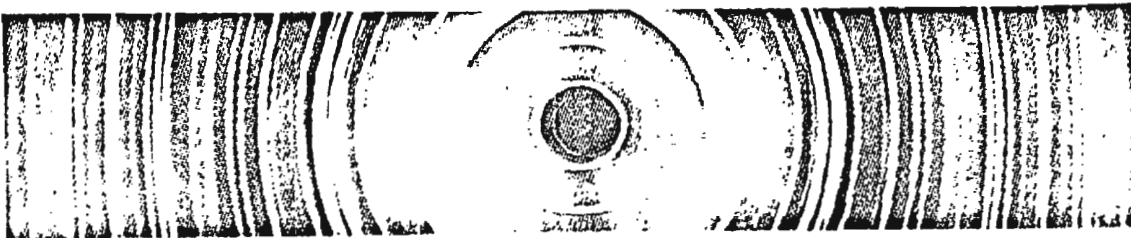
Patterns not included: Calcite (I34), Muscovite (I35),
Tremolite (I19/I20), Magnesite (I6),
Dolomite

Most probable minerals present: Talc, Chlorite

Visual Comparison: Talc, Chlorite

Minerals Present: Talc, Chlorite

J&J-0130652

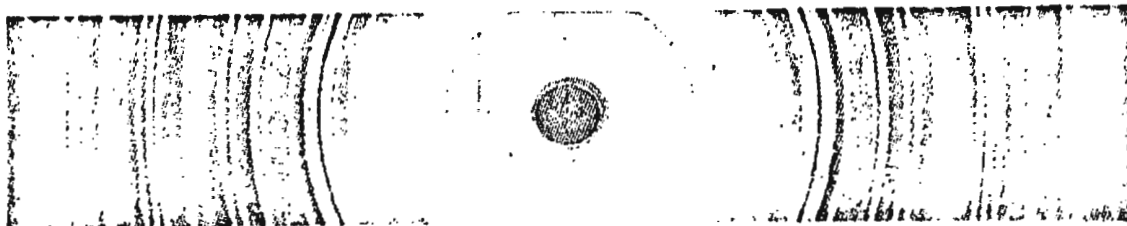
SAMPLE I42 COLOURED TALC No.1.Comparison

Patterns not included: Magnesite (I37), Talc (I46), Muscovite (I35), Dolomite.

Most probable minerals present: Chlorite, Talc.

Visual Comparison: Chlorite, Talc

Minerals Present: Chlorite, Talc

SAMPLE I43 FIBROUS TALC FACE 10

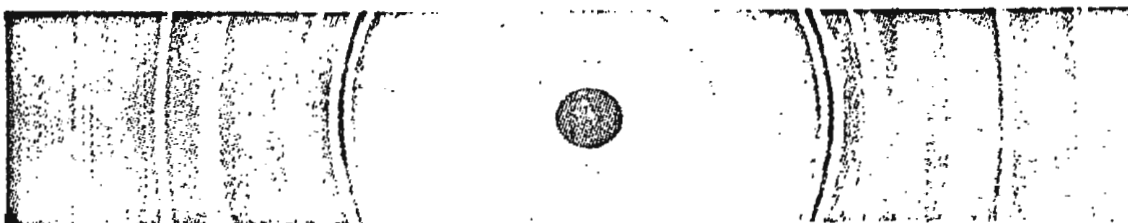
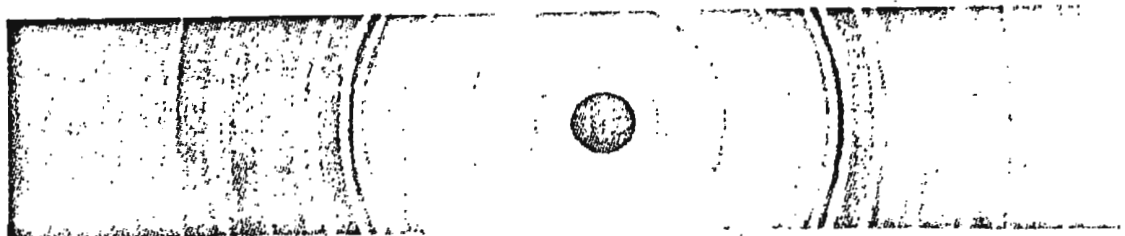
Comparison: 2 unmatched lines $4.8928\overset{\circ}{A}$, $4.4431\overset{\circ}{A}$

Patterns not included: Calcite (I34), Magnesite (I37), Muscovite (I35), Tremolite (I19/I20)

Most probable minerals Present: Chlorite, Talc

Visual Comparison: Chlorite, Talc

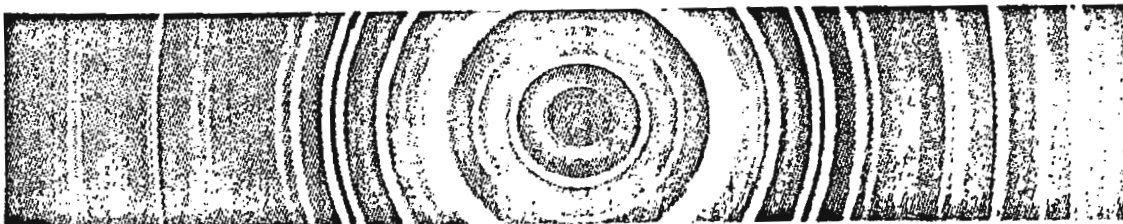
Minerals Present: Chlorite, Talc

SAMPLE I44 PURE TALC FACE 1Comparison: 1 unmatched line 1.0798Patterns not included: Magnesite (I37), Talc (I42),
Muscovite (I35), Tremolite (I19/I20)Most probable minerals present: Chlorite, Talc, DolomiteVisual Comparison: Talc, Magnesite, ChloriteMinerals Present: Talc, Magnesite, ChloriteSAMPLE I45 GOOD SPECIMEN FACE 1Comparison: 2 unmatched lines 1.0882^oA, 1.0505^oAPatterns not included: Calcite (I34), Chlorite (I42), Chlorite (I4)
Muscovite (I35), Magnesite (I6), Tremolite (I19/I20), Dolomite.Most probable minerals present: Talc, MagnesiteVisual Comparison: TalcMinerals Present: TalcSAMPLE I46 COLOURED TALC FACE 3Comparison:Patterns not included: Chlorite (I42), Chlorite (I4), Muscovite
(I35), Tremolite (I19/I20).Most probable minerals present: Talc, MagnesiteVisual Comparison: Talc, MagnesiteMinerals Present: Talc, Magnesite

Specimen Patterns and Comparison Data for
Samples of Old Powders and ~~00000~~ Shipments

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SAMPLE BATCH 6 POWDER F1 PW.J. 035Comparison: 1 unmatched line 8.1972⁰ÅPatterns not included: Muscovite (I35), Tremolite (I19/I20)Most probable minerals present: Talc, Magnesite, ChloriteVisual Comparison: Talc, Chlorite, MagnesiteMinerals Present: Talc, Chlorite, MagnesiteSAMPLE BATCH 8 POWDER (S and G) PW.J. 035ComparisonPatterns not included: Magnesite (I6), Tremolite (I19/I20)Most probable minerals present: Talc, Magnesite, Boric AcidVisual Comparison: Talc, Chlorite, Boric AcidMinerals Present: Talc, Chlorite, Boric AcidSAMPLE BATCH 9 POWDER T4 P.W.J. 035Comparison: 1 unmatched line 1.2587⁰ÅPatterns not included: Tremolite (I19/I20)Most probable minerals present: Talc, Chlorite, muscovite,
Magnesite, Boric AcidVisual Comparison: Talc, Chlorite, Boric AcidMinerals Present: Talc, Chlorite, Boric Acid

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JNJAZ55_00001767

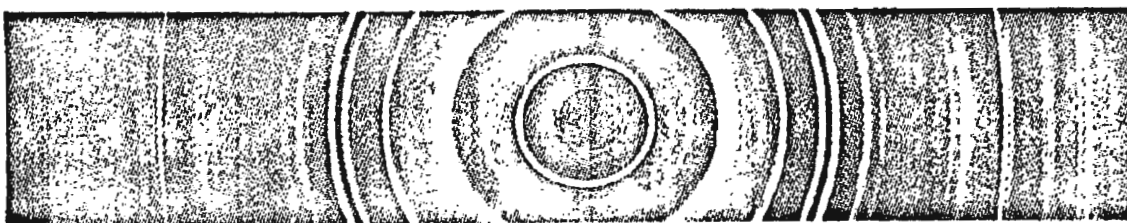
SAMPLE BATCH 10 POWDER SK1BP PW.J. 035Comparison

Patterns not included: Calcite (I34), Muscovite (I35),
Tremolite (I19/I20), Dolomite

Most probable minerals present: Talc, Chlorite, Magnesite,
Boric Acid.

Visual Comparison: Talc, Chlorite, Boric Acid

Minerals Present: Talc, Chlorite, Boric Acid

SAMPLE BATCH 11 POWDER LD18P PW.J. 035

Comparison: 1 unmatched line 8.1363⁰_A

Patterns not included: Magnesite (I6), Tremolite (I19/I20)
Dolomite

Most probable minerals present: Talc, Chlorite, Boric Acid

Visual Comparison: Talc, Chlorite, Boric Acid, Magnesite

Minerals Present: Talc, Chlorite, Boric Acid, Magnesite

SAMPLE BATCH 12 TALC 1960 PW.J. 025

Comparison: 1 unmatched line 8.12⁰_A

Patterns not included: Tremolite (I19/I20)

Most probable minerals present: Talc, muscovite, chlorite,
Boric Acid.

Visual Comparison: Talc, Chlorite, Boric Acid, Magnesite

Minerals Present: Talc, Chlorite, Boric Acid, Magnesite

SAMPLE BATCH 13 TALC 1961 PW.J. 026Comparison

Patterns not included: Calcite (I34), Muscovite (I35)
Tremolite (I19/I20)

Most probable minerals present: Talc, Chlorite, Magnesite
Boric Acid

Visual Comparison: Talc, Chlorite, Magnesite, Boric Acid

Minerals Present: Talc, Chlorite, Magnesite, Boric Acid

SAMPLE BATCH 19 S.S. CATHERINA W. 02/05/72Comparison

Patterns not included: Tremolite (I19/I20)

Most probable minerals present: Talc, Chlorite, Magnesite

Visual Comparison: Talc, Chlorite, Magnesite

Minerals Present: Talc, Chlorite, Magnesite

SAMPLE BATCH 2 TALC S.S. EDNA 'B' 14/02/72Comparison

Patterns not included: Talc (I45), Tremolite (I19/I20)

Most probable minerals present: Talc, Chlorite

Visual Comparison: Talc, Chlorite

Minerals Present: Talc, Chlorite

CONCLUSIONS

The optical examination has shown that there are a large number of minerals associated with the rock types found both in the talc seam and in the associated rocks. The footwall rocks in contact with the talc are mainly composed of the minerals quartz, muscovite, chlorite, garnet, and some carbonate material both calcite and magnesite. Minor minerals in the footwall contact rocks include epidote, microcline, tremolite and actinolite, sphene, rutile, hornblende, rare talc, biotite, pyrite, pyrrhotite and chalcopyrite. Rock type inclusions into the talc have similar compositions to the footwall rocks but with higher muscovite and chlorite contents. The muscovite was generally an iron rich variety (phengite), while two forms of chlorite were observed namely sheridanite and penninite. Other talc inclusions consist mainly of carbonate minerals, calcite and magnesite in varying quantities. It is with these nodules that some tremolite is found. The rocks further away from the talc seams, namely the gneiss, become richer in quartz and microcline and below these marble occurs.

The carbonate specimens examined by optical means showed that the carbonate minerals, calcite and magnesite, were accompanied by talc, chlorite, tremolite, muscovite, rutile and pyrite, all in minor amounts. In general the carbonate inclusions were large and very discrete in the talc seam itself.

The specimens examined, which can be classified as talc samples, were found to be in the main composed of talc with chlorite as the major contaminant. Some specimens, however, were predominantly composed of chlorite with minor talc inclusions. Other minerals found in association with the talc specimens included garnet, rutile and magnesite with rare tremolite and a quartz or serpentine inclusion. Some differences were observed in the talc itself, some of the talc appearing to be a little murky in texture. X-ray pictures of the clear and murky material showed no differences however.

The powder X-ray examination confirmed the major minerals occurring in the hand specimens and a classification was possible into the three groups already mentioned, i.e. rock types, carbonate samples and talc specimens. The only asbestos type mineral to be detected in the hand samples was tremolite, which was found in three of the specimens. The tremolite was associated with carbonate minerals, namely magnesite and calcite, no tremolite was detected in the talc type specimens. Chlorite was, however, very common in the talc types, some of the specimens being very nearly pure chlorite in composition. There appeared to be some association of the chlorite with coloured talc specimens, especially those with a greyish colour. Other colour variations due to rutile were not detected by X-ray examination.

The examination of consecutive samples at face 1 in the mine showed that the chlorite content can vary very drastically over a 6ft thick section of the talc seam. Patterns obtained from several shipments of ~~000000~~ talc showed that chlorite, together with carbonate material, were the major contaminant minerals. This was also true of powder samples ranging back to 1949 in which the only observable difference was the presence of boric acid.

The electron microscope examination of the powdered samples showed that a difference could be drawn between particles produced from the various samples. The carbonates and rock types on the whole produced compact fibre free particles. The talc specimens were, however, plate-like in appearance with varying quantities of lath like particles coupled with fibres which were textile in appearance. Both lath and textile types of particles were not composed of minerals associated with the commercial asbestos industry. Particles formed from the amphibole mineral found at the mine were hardly fibrous in character, the majority of the tremolite breaking to give compact particles. Those fibres formed were short and had a very large diameter when compared with the commercial varieties of asbestos. No amphibole or chrysotile mineral was detected in any of the numerous powders examined.

The Italian talc ~~000000~~ contains observable quantities of chlorite and carbonate minerals and could contain any one of the following minerals in very minor amounts: muscovite, quartz, tremolite, garnet and rutile. If small pieces of footwall rock were to contaminate the ore during production, several of the other listed minerals found in the rock type specimens could appear in the shipped product. It is unlikely that they would be present in detectable amounts.

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Project Supervisor

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J&J-0130661

JNJAZ55_000017681

HARWELL

In confidence

J&J-0130662

JNJAZ55_000017682

COMMERCIAL IN CONFIDENCE

AERE-G63

The Morphology and
Characterization of Tale

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Materials Development Division,
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August, 1972

J&J-0130663

Synopsis

Samples of talc submitted by Johnson and Johnson Ltd. have been examined by a number of physical techniques to characterize the powder and identify any impurities. Examination in both the scanning electron microscope and the transmission electron microscope suggested the possible presence of a few rod-shaped particles as well as the normal platelets but non-dispersive analysis of the X-ray spectra and electron diffraction failed to differentiate between the particles.

X-ray powder diffraction data suggested the presence of a material other than talc in amounts less than 1%. The extra diffraction peaks could not be accounted for completely by published data in the Powder Diffraction File and the most satisfactory conclusion was that the bulk of the impurities were present as Bavalite.

*The talc samples supplied to A.E.R.E. were
of regular Italian talc ex. Vail Chisone
mine. Grade 00000*

*J. R. Dean
Director of Research
Johnson & Johnson U.K. Ltd.
25.4 Aug 72*

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3. Scanning electron microscopy
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 - 4.2 Morphology
 - 4.3 Electron diffraction
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 - 5.2 Attempts to identify additional reflections
6. Electron microprobe analysis
7. Discussion
8. Conclusions
9. Acknowledgements

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Figure 3 Scanning electron micrograph x 10,000
Figure 4 Transmission electron micrograph x 10,000
Figure 5 Electron microprobe scan for calcium
Figure 6 Electron microprobe scan for aluminium in same area as for Figure 5

1. Introduction

Samples of commercially processed talc (magnesium silicate) from their Italian source were received from Johnson and Johnson Ltd. for a thorough characterization together with identification of any natural impurities. After preliminary X-ray diffraction studies had suggested that material other than talc was present at the sub-1% level, samples of chlorite bedrock and magnesite inclusions taken from the Italian mine were supplied as aids to identification.

The various physical methods of examination used were optical microscopy, scanning electron microscopy including non-dispersive analysis, transmission electron microscopy, electron diffraction, X-ray powder diffraction techniques and electron microprobe analysis.

2. Optical Microscopy

A sample of the powder was lightly dusted on to a glass slide and examined under low power binoculars and in a metallurgical microscope. As expected, the majority of the particles were plate-like but a few were observed in the form of rods or needles. In contrast to the majority of the particles which had a whitish lustre, a few were observed with a light green tinge. However, the particle size was too small for adequate resolution optically and it was clearly necessary to resort to electron microscopy to characterize the particles satisfactorily.

3. Scanning Electron Microscopy

3.1 Preparation of Sample

Some powder was dusted on to a specimen stub that had been coated with a colloidal suspension of graphite in alcohol. This gave a thin layer of particles without the formation of large agglomerates. A conducting layer of carbon was subsequently deposited by evaporation on the particles.

3.2 Topographical examination

Examination in the Stereoscan at magnifications up to about $\times 10,000$ generally confirmed the results of the optical work. Platelets were predominantly present in various orientations and some needle-like configurations were clearly accounted for by plates which were viewed in an 'edge-on' position and this was confirmed by taking stereographic photographs. Typical structures obtained are illustrated in Figures 1, 2 and 3.

3.3 Chemical analysis using Non-Dispersive Techniques

Non-dispersive X-ray analysis was carried out on a number of particles using a lithium drifted silicon solid state detector. No significant

variation was found in relative intensity of the silicon and magnesium characteristic emissions suggesting that they were all of similar relative composition. This does not, however, prove that all the particles analysed were talc for the following reasons:- i) there is no guarantee that all the X-ray emission measured was from an individual platelet and ii) it does not exclude the presence of an impurity of similar magnesium/aluminium ratio to that of talc. The X-ray spectra obtained did not show significant emissions of other metallic elements indicating the absence of any major impurity.

4. Transmission electron microscopy

4.1 Preparation of sample

An equal volume of talc powder and a solution of 15% nitrocellulose in amyl acetate was rubbed on to a glass plate with a spatula. This aliquot was then diluted with acetone and spread over a clean microscope slide to dry. The dried film was scored into 3mm square sections which were floated off the slide on to water and picked up with an electron microscope grid. After drying off on a filter paper, a supporting film was then evaporated over the surface. This yielded a nitrocellulose film in which the talc platelets were encapsulated with their planes lying generally in that of the film to preclude 'edge-on' effects.

4.2 Morphology

The platelets in the film were examined in an EM300 transmission microscope at magnifications of up to x 80,000. This revealed that the rod-shaped particles which were about 250 Å diameter and varied in length up to 0.15µm, could have been platelets which had folded or rolled up, but this was not positively confirmed (see figure 4).

4.3 Electron diffraction

Electron diffraction patterns were obtained in situ in the microscope from a number of particles with both types of appearance but no real evidence was obtained of a structural difference between the plates and "rods". Two diffraction patterns showed evidence of preferred orientation but this was to be expected from their nature and was not considered to be indicative of any chemical difference between the two particles. It must be noted, however, that although the method of preparing the sample for examination had been chosen to separate the particles as much as possible, there was difficulty in guaranteeing that completely isolated particles had been selected for diffraction.

5. X-ray diffraction

5.1 Experimental

Some of the powder was packed in silica capillary tubes for both X-ray diffraction patterns and diffractometer traces. The results showed absence of preferred orientation and this was probably due to the platelets aligning themselves in the silica tube when tapped in the usual way to concentrate them in the bottom portion. Patterns were obtained from two completely different batches of talc supplied by the firm and also from two mineral samples said to be Magnesite and Chlorite, although the latter was subsequently discovered to be Muscovite, a micaceous mineral.

The same reflections were obtained from both talc samples but some differences were observed in the relative intensities of some reflections. Since some preferred orientation was evident in both patterns, these intensity differences were most likely due to slight variations in the type and/or degree of preferred orientation of the platelets in the two samples. The overall conclusion was that the two talc samples were identical in nature and that any impurity was due to the same compound or compounds.

The first sixty six reflections from the patterns were selected for identification and the calculated d-spacings are listed in Table I. Of these, all but 27 were satisfactorily accounted for by the available data on talc published in the Powder Diffraction File. This strongly suggested that an impurity was present to an extent judged to be below the 1% level. The strongest reflections from the samples of Magnesite and Muscovite were absent in our talc patterns so that neither could reasonably account for the impurity reflections.

A check was made of the possibility that the impurity was Tremolite but none of the extra reflections agreed with the published data for this mineral. There are a wide range of minerals in the Chlorite group which is a distinct possibility as an impurity since their crystal structure is related to that of talc and brucite. X-ray diffraction data have been published on five varieties and these were checked with the talc patterns. In the case of Bavalite (a variety of Daphnite), it was found that 24 of our 66 reflections could be accounted for by this form of Chlorite, although 16 of these were also included in the published data for talc. Bavalite can contain traces of alkaline earths, copper, titanium or manganese but the sample from which the X-ray data were obtained was reported to be $(\text{Mg}_{0.4}\text{Fe}_{4.2}\text{Al}_{1.5})(\text{Si}_{2.6}\text{Al}_{1.4})\text{O}_{10.2}(\text{OH})_{7.8}$.

5.2 Attempts to identify additional reflections

In attempts to prove or disprove that the extra 27 reflections were all accounted for by a small percentage of impurity, a number of approaches were investigated. A thorough computer search of the Powder Diffraction File was made to list possible compounds which could account for the unidentified reflections. Of a whole variety of possibilities including both inorganic and organic compounds, the only three which appeared at all possible were Bavalite, potassium dizirconate $K_2Zr_2O_5$ and calcium aluminium oxide hydrate, $CaAl_2O_4 \cdot 10H_2O$. In the case of the dizirconate, the talc patterns account for only two low angle lines of medium intensity and other stronger lines are not present. The two strongest lines of the calcium compound could be accounted for by two low angle lines in our patterns plus two others at higher angles (where talc reflections also occur) but that would still leave eleven lines unidentified. On the basis of published X-ray data therefore, Bavalite appeared to be the most likely impurity.

Since both the talc patterns were consistent with some preferred orientation being present in the powder samples, it was considered that some of the extra reflections might have been due to enhancement of very weak reflections from talc which would not be observed in the standard pattern from a perfectly random sample. A computer calculation was made of all possible reflections from talc assuming that the published crystal structure was correct and on this basis, a further 16 of the 27 unknown reflections in the talc patterns could be satisfactorily indexed.

6. Electron Microprobe Analysis

As the X-ray diffraction data strongly suggested the presence of an impurity which could not be identified unambiguously as Bavalite, a limited amount of electron microprobe analysis was considered worthwhile. The sensitivity of this instrument for detecting impurity elements is significantly better than that of the scanning electron microscope and these could have been missed in the work described in Section 3.3. The seven elements selected as being most representative of the likely impurity materials were fluorine, potassium, calcium, nickel, aluminium, iron and chromium. The last four of these might be expected if some varieties of Chlorite were present, calcium for Tremolite, calcium and iron for Actinolite and calcium, fluorine and potassium for Apophyllite. The presence of iron and aluminium would substantiate the possibility of Bavalite.

A sample of the talc was sprinkled on a bakelite disc to give as even a layer as possible and a search was made for each of the seven elements listed. Although the level of the characteristic X-rays for iron was very close to that of the background, there was a possibility that iron was present in a very low concentration throughout the sample. A few discrete areas were found in which calcium and aluminium were detected together as shown in Figures 5 and 6 but the origin of the characteristic X-rays from these two elements could not be traced to individual particles as the lateral and depth resolution of the electron beam was much greater than the average particle size. None of the elements fluorine, potassium, nickel and chromium were present at the level of sensitivity which was about 100 ppm. The results were therefore inconclusive although not inconsistent with the possibility that Bavalite was the major impurity.

7. Discussion

The main evidence for the presence of an impurity in the talc samples comes from the X-ray diffraction data which shows some 27 reflections in addition to those included in the published information. Just over half of these extra reflections can be accounted for by assuming that some weak reflections, not normal of significance for a randomly oriented sample, are enhanced due to preferential type of packing the flakes in the silica tube. This still leaves some reflections completely unaccounted for by talc, the strongest of which agreed with the published data for Bavalite. This mineral is said to occur in the form of sheets and to be greenish in colour which would be consistent with the observations in the optical microscope. Since neither the transmission or scanning electron microscopy was able to distinguish between the rod like particles and the platelets, it seems probable that these were folded platelets as postulated. The limited electron microprobe analysis carried out indicated that calcium and aluminium occurred together at the impurity level in the powder and that traces of iron were also present. This would also be consistent with the presence of a small concentration of Bavalite if some of the magnesium were replaced by calcium as is known to occur in some varieties. Other possible explanations of the extra reflections in our X-ray powder diffraction data are (a) that the published data on mineral talc is incomplete or (b) that small concentrations of impurities such as calcium and aluminium are present in the talc and give rise to the additional lines by slightly modifying the crystal structure. Neither explanation seems acceptable and the possibility of Bavalite being present in an amount less than 1% appears more likely and to fit all the observations.

8. Conclusions

The two samples of talc examined are essentially the same and contain a common impurity present in an amount less than 1%. The extra reflections in the X-ray powder pattern not accounted for by talc are consistent with the impurity being one form of Chlorite known as Bavalite.

9. Acknowledgements

The work reported was carried out by various members of the Solid State Instruments Group whose suggestions and comments are greatly appreciated.

Circulation

Messrs. Johnson and Johnson Ltd. (5 copies)
Commercial Office
A.E.R.E. Library
Mr. B. W. Mott

Table I X-Ray Powder Diffraction Data

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Data from Diffractometer Trace		Data from Powder Diffraction File			
d spacings (observed)	Intensity % of strongest	P.D.F. 19-770 Talc		P.D.F. 7-166 Bavalite	
		d	I	d	I
** 13.9846	13			14.0	60
9.3585	100	9.35	100		
** 8.6130	31				
** 8.1539	7				
** 7.4367	2				
** 7.0418	44			7.08	100
** 6.5298	1				
4.6756	84	4.67	8	4.681	30
4.5428	11	4.56	25		
4.3218	8	4.33	6		
* 4.2462	1				
4.1235	3	4.12	6		
3.8750	1	3.87	2		
3.5366	49	3.52	< 2	3.523	50
* 3.4386	3				
* 3.3337	36				
* 3.2867	3				
* 3.2456	4				
* 3.1921	20				
3.1125	89	3.12	40		
* 2.8883	37				
* 2.8330	18			2.821	10
** 2.7993	1				
* 2.7279	4				
2.6254	2	2.627	8	2.619	30
2.5887	5	2.589	14	2.574	20
* 2.5321	1				
2.4747	10	2.479	30	2.469	10
* 2.4200	2			2.405	20
2.3394	36	2.337	2		
2.2769	1	2.289	2	2.279	10
2.2116	3	2.219	6		
2.1910	3	2.188	2		
2.0987	1	2.086	2		
2.0668	1	2.071	2		

J&J-0130672

JNJAZ55_000017692

Data from Diffractometer
TraceData from Powder Diffraction
File

	d spacings (observed)	Intensity % of strongest	P.D.F. 19-770		P.D.F. 7-166	
			Talc d	I	Bavalite d	I
*	2.0281	17			2.014	20
*	1.9997	1				
	1.9786	1	1.967	2		
	1.9179	1	1.921	2	1.893	5
	1.8697	71	1.870	4		
*	1.8173	1			1.828	5
*	1.7857	1			1.765	5
	1.7338	2	1.731	10	1.722	5
	1.6879	7	1.684	6		
	1.6715	8	1.668	6		
	1.6529	4	1.654	2	1.666	5
*	1.5777	1				
	1.5573	56	1.558	2	1.560	20
	1.5418	1	1.540	< 2		
	1.5268	4	1.529	55	1.523	10
**	1.4764	1			1.483	5
**	1.4650	2				
**	1.4473	2				
**	1.4338	5			1.427	5
	1.4145	7	1.411	4	1.411	5
	1.3958	12	1.386	4	1.393	5
	1.3858	12	1.376	2		
	1.3354	48	1.336	2	1.339	5
	1.3179	1	1.318	8		
	1.2945	2	1.295	10		
	1.2500	1	1.255	2		
	1.2411	2	1.243	2		
	1.2383	2	1.233	2		
	1.2230	6	1.219	< 2	1.228	5
	1.1828	1	1.187	4		
	1.1688	10	1.169	2		

* Accountable on computer-calculated pattern for TALC

** Unaccountable on basis of TALC

N.B. Calculated TALC pattern did not extend below d = 1.600



FIGURE 1. Scanning Electron Micrograph

x 500



FIGURE 2. Scanning Electron Micrograph

x 500

J&J-0130674



FIGURE 3. Scanning Electron Micrograph

x 10,000



FIGURE 4. Transmission Electron Micrograph

x 10,000

J&J-0130675



FIGURE 5. Electron Microprobe Scan for Calcium

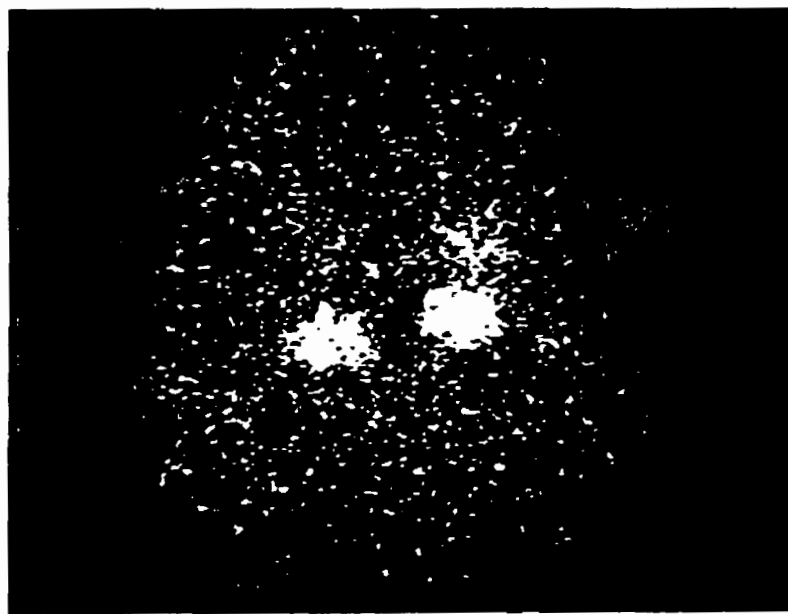


FIGURE 6. Electron Microprobe Scan for Aluminium in Same Area as Above

J&J-0130676

JNJAZ55_000017696

Mining Institute
Torino, Italy

J&J-0130677

PETROGRAPHIC IDENTIFICATION OF ASBESTOS SILICATES IN VAL CHISONE
(SVC) TALC.

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by The Mining Institute of
The Polytechnical School of Torino.

The analysis was performed on two samples of SVC pulverized talc produced by two different mills in different times.

Operation: Small amounts of the samples have been dispersed in eugenol and observed at the microscope under 5 different conditions: phase contrast, dark field, bright field, polarized light (parallel and crossed nicols).

RESULTS:

The samples, though rather different in general composition are similar in regard to the content of particles morphologically correspondent to asbestos minerals.

No fibrous particles having the distinguishing features of serpentine asbestos (chrysotile) occur in the samples.

There are no particles having the optical features of amphibole asbestos like amesite or crocidolite.

The only particles that occasionally occur are those ones showing the optical features of tremolite. Their proportions range in number between 1 and 2 per 10,000 parts; that means a weight content less than 0.02 %.

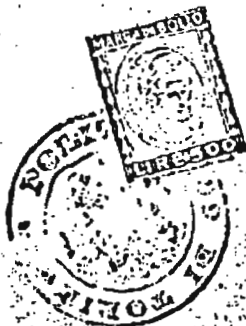
This last result has been backed by a Vreeland spectro-scope test on the same samples after they were purified of calcium and magnesium carbonates. Calcium, which has a proportion content of 9.9 % in the tremolite formula, was found close to the lowest limit of sensitivity of the spectroscope (about 0.01 %).

Torino, February 1972

J&J-0130678



POLITECNICO DI TORINO
ISTITUTO DI ARTE MINERARIA



10129 - TORINO, 16 febbraio 1972
Corso Duca degli Abruzzi, 24
Centralino Politecnico 53 16 16 int. 287
Telefono: Istruttoria (linea diretta) 53 39 96

N.	Risposta a	del
16/G3		

Relato: CERTIFICATO N. 71/7211/

DETERMINAZIONE PETROGRAFICA DI SILICATI ASBESTOIDI

Committente: Società Talco e Grafite Val Chisone - Pinerolo (Torino)

Richiesta: lettera VA/rb. P.S. del 4/2/1972

Campioni: N. 2, contrassegnati con le sigle "SVC Extra 00000 San Sebastiano" e "SVC Extra 00000 Malannaggio" consegnati personalmente all'Istituto da parte dell'Ing. Sartorio, Direttore Tecnico della Committente, in data 4/2/1972.

Determinazione richiesta: "dosaggio del quantitativo di tremolite e di analoghi silicati asbestici".

Modalità di analisi: Una porzione rappresentativa dei campioni in esame è stata dispersa in eugenolo ed osservata al microscopio in 5 differenti condizioni: contrasto di fase, campo oscuro, campo chiaro, luce polarizzata (a nicols paralleli ed incrociati)

Risultati delle determinazioni

1. - I due campioni, sebbene lievemente differenti come composizione generale, sono del tutto equivalenti nei riguardi del contenuto di elementi bacillari, di caratteristiche morfologiche corrispondenti ai minerali asbestoidi;
2. - Nei campioni non si riscontrano elementi fibrosi aventi le caratteristiche dell'amianto di serpentino (crisotilo);

L'operatore:

Souley
IL DIRETTORE AMMINISTRATIVO

Visto:

IL DIRETTORE
ISTITUTO DI ARTE MINERARIA
[Signature]

J&J-0130679

JNJAZ55_000017699

- 3 - Non è presente alcun individuo dotato delle caratteristiche ottiche degli amianti d'anfibolo di tipo amosite o crocidolite;
- 4 - Sono presenti rari elementi bacillari aventi le caratteristiche ottiche della tremolite, in proporzioni corrispondenti a circa 1-2 parti per 10000 elementi (in numero); tenuto conto della forma degli elementi stessi, il contenuto ponderale è certamente inferiore allo 0,02%.

Osservazioni:

- 1) Una prova di carattere minerogenetico dell'assenza dell'amianto crisotilo è data dall'incompatibilità paragenetica di tale amianto con le rocce talcose;
- 2) Una prova analitico-strumentale del ridottissimo contenuto di tremolite è costituito dal saggio spettroscopico alla fiamma sui campioni previamente epurati dai carbonati di calcio e magnesio per debole attacco acido: le linee corrispondenti al calcio (elemento contenuto nella tremolite in proporzioni stechiometriche del 9,9%) si ritrovano presenti ai limiti di sensibilità dello spettroscopio di Vreeland utilizzato per l'indagine (pari allo 0,01% circa).

L'operatore:

(Prof. Ing. Enea Occella)

Enea Occella

Visto:



ISTITUTO DI ARTE MINERARIA
DIRETTORE

[Signature]

DIRETTORE AMMINISTRATIVO

[Signature]

Prof. S. Lewin

J&J-0130681

JNJAZ55_000017701

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Importers

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REPRESENTATIVE IN U.S.A. OF FOREIGN MINES

September 18, 1972

Johnson & Johnson
New Brunswick, New Jersey 08903

Att: Mr. Harold Hogan

Dear Harold:

Confirming our telephone conversation regarding the source of the basic rock from which 5/0 Italian Talc and #1615 AGI Italian Talc are made.

Both processed products are made from the same basic rock and this rock is obtained from the Fontana mine located at Val Germanesca in the Italian Alps of Northern Italy.

No. 5/0 product is ground and packaged for shipment in Italy. As you know, your Company has not been using any of this product for a number of years.

No. 1615 AGI is ground and packaged in the United States at the Metropolitan Talc Company plant located in South Plainfield, New Jersey.

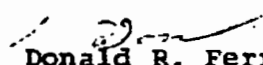
This plant is jointly owned by Charles Mathieu, Inc. and Whittaker, Clark & Daniels, Inc. This is the product your Company has been purchasing and is also the product covered by the attached analysis confirming the absence of any asbestos.

Our Quality Control procedure at the Metropolitan Talc Co. calls for complete cleaning and checking before any changes of feed stock (rock) are packaged for shipment. If you require further information regarding our products, please do not hesitate to call.

Very truly yours,

CHARLES MATHIEU, INC.

DRF:as


Donald R. Ferry

Not responsible for damages incurred by carriers. All quotations and contracts subject to conditions beyond our control.
Prices are based upon existing tariff rates and customs regulations.

J&J-0130682

JNJAZ55_000017702

Final Report of X-Ray Diffraction Analysis*= 5th Italian*

Sample: No. 1615, Whittaker, Clark, and Daniels
(Talc, AGI)

Mineral Constituents: a) 85% Talc ($\pm 2\%$)
10% Chlorite ($\pm 2\%$)
2% Dolomite ($\pm 2\%$)
1-2% Quartz ($\pm 2\%$)
2% Phlogopite ($\pm 2\%$)

b) No detectable amounts of any of the
asbestiform minerals.

Nov. 2, 1971

Seymour Z. Lewin
Seymour Z. Lewin
Professor of Chemistry

Final Report of X-Ray Diffraction Analysis

Sample: Italian Talc Rock
(Hand Specimen)

Mineral Constituents: 90% Talc ($\pm 2\%$)
5% Chlorite ($\pm 2\%$)
5% Phlogopite ($\pm 2\%$)

Nov. 2, 1971

Seymour Z. Lewin
Seymour Z. Lewin
Professor of Chemistry

J&J-0130684

JNJAZ55_000017704

J&J-0130685

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Prof. M. Buerger

J&J-0130686

JNJAZ55_000017706

Martin Julian Buerger

Academic Training

S.B., Mining Engineering, M.I.T., 1925

S.M., Geology, M.I.T., 1927

Ph.D., Mineralogy, M.I.T., 1929

Academic Positions, Massachusetts Institute of Technology

Assistant in Geology, 1925-1927

Instructor in Geology, 1927-1929

Assistant Professor of Mineralogy and Petrology, 1929-1935

Associate Professor of Mineralogy and Petrology, 1935-1944

Professor of Mineralogy and Crystallography, 1944-present

Institute Professor, 1956-present

Chairman of the Faculty, 1954-1956

Director of the School for Advanced Study, 1956-1963

Academic Positions, University of Connecticut

University Professor, Department of Geology, 1968

Graduate Advisor, Institute of Materials Science, 1968

Society Membership

American Crystallographic Association

Geological Society of America (Fellow)

Mineralogical Society of America (Fellow)

National Academy of Sciences, U.S.A., 1953

Honorary Membership, foreign societies

Academia Brasileira de Ciencias, 1952

Accademia delle Scienze di Torino, 1954

Deutsche Mineralogische Gesellschaft, 24. August, 1957

Accademia Nazionale dei Lincei, 1960

Bayerische Akademie der Wissenschaften (corresponding), 1960

Osterreichische Akademie der Wissenschaften (corresponding), 1962

Real Sociedad Española de Historia Natural of Madrid, March, 1964

Offices Held

Societies

President, American Crystallographic Society, 1939-1946

President, American Society for X-ray and Electron Diffraction,
1948

President, Mineralogical Society of America, 1947

Vice-President, Geological Society of America, 1948

Councilor, American Academy of Arts and Sciences, 1950-1954

International Union of Crystallography

American Delegate to Preliminary Convention, 1946

Council Member, 1946-1951

Co-Editor, International Tables for X-ray Crystallography,
1946-present

Member, Apparatus Commission, 1951-1956

U.S.A. National Committee on Crystallography, 1951, 1952, 1956-1962,
1966-1968

National Research Council

Representative of the U.S.A. to the Second General Assembly
and Congress of the International Union of
Crystallography, Stockholm, 1951

Representative of the U.S.A. to the Fourth General Assembly
and Congress of the International Union of
Crystallography, Cambridge, England, 1960

Member, Committee on Solids, 1949-1953, 1956-1962

Zeitschrift für Kristallographie

Co-Editor, 1953-present

Faculdade de Filosofia, Universidade de Rio de Janeiro

Visiting Professor, 1948

Visiting Professor. Consejo Superior de Investigaciones Cientificas,
Madrid, 1958

Instituto de Física y Matemáticas, Universidad de Chile,
Guest Lecturer, 1962.

Visiting Professor, University of Minnesota, 1970

Visiting Professor, University of Kentucky, 1971

J&J-0130688

Honors

Day Medalist, Geological Society of America, 1951.
 Roebling Medalist, Mineralogical Society of America, 1958
 D.h.c., University of Bern, 1958
 Isador Fankuchen Awardee, American Crystallographic Society, 1971

Books

The Optical Identification of Crystalline Substances.
 (The M. I. T. Letter Shop, Cambridge, 1939)
 X-ray Crystallography. (John Wiley and Sons, Inc., New York, 1942)
 Elementary Crystallography. (John Wiley and Sons, Inc.,
 New York, 1956)
 The Powder Method. (with Leonid V. Azaroff) (McGraw-Hill Book Co.,
 Inc., 1958)
 Vector Space. (John Wiley and Sons, Inc., New York, 1959)
 Crystal-Structure Analysis. (John Wiley and Sons, Inc., New York,
 1960)
 The Precession Method. (John Wiley and Sons, Inc.,
 New York, 1964)
 Contemporary Crystallography (McGraw-Hill, New York, 1970)
 Introduction to Crystal Geometry (McGraw-Hill, New York, 1971)
 The Laue Method (1973)

DEPARTMENT OF EARTH AND PLANETARY SCIENCES

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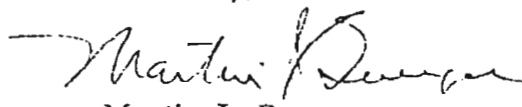
MASSACHUSETTS INSTITUTE OF TECHNOLOGY
CAMBRIDGE, MASSACHUSETTS 02139

7 October 1972

Dear Dr. Goudie,

I am enclosing a face copy and three Xerox copies of my report on the Washington meeting, which you requested in your letter of September 25. I have also taken the liberty of including some biographical material on myself in order to strengthen your hand if it is necessary to defend the authority of some of the remarks I make concerning the results of the meeting. In particular, I would like to draw your attention to the first book I ever wrote which you will find on the last page of the biographical information. I taught this subject for perhaps 20 years, so I feel that the remarks made concerning Lewin's identification of chrysotile can be justified.

Sincerely,



Martin J. Buerger

Dr. A. L. Goudie,
Johnson & Johnson Research Center,
New Brunswick,
New Jersey.

J&J-0130690

JNJAZ55_000017710

In the following report (first paragraph, page 2), Professor Buerger's statement indicating the use of step scanning by Professor Gordon Brown and Colorado School of Mines Research Institute should be corrected to Johnson & Johnson and Colorado School of Mines Research Institute.

The Washington Meeting of September 21, 1972

of

Johnson and Johnson

with the

Food and Drug Administration

and

Conclusions to be drawn from the meeting

by

Martin J. Buerger

October 7, 1972

J&J-0130692

As a preliminary to the meeting with the Food and Drug Administration, those associated with Johnson & Johnson met early on the afternoon of the previous day, September 20, in the Holiday Inn, to review their findings. The Johnson & Johnson staff included Dr. Fuller, Dr. Shelley, Dr. Rollé, Jack Sheltz and Dr. Nashed. Associates of Johnson & Johnson included Walter McCrone and Ian Stewart, Fred Pooley, Sidney Pollack, Gordon Brown and Martin Buerger. These reported to the other members of the group the results of their investigations of the purity of the talc used in the Johnson & Johnson talcum powder known as "Shower-to-Shower". The general conclusion was that none of the work revealed any trace of contamination of "Shower-to-Shower" talc by the mineral chrysotile.

The meeting of the Johnson & Johnson group with the Food and Drug Administration took place September 21, 1972, on the 6th floor conference room of 200 Avenue C, S.W., Washington D.C. It was chaired by Dr. Schaffner, who introduced, before the meeting, Messrs. Weissler, Winniger, Carolla and a number of visitors whose names were not clearly given. Professor Seymour Lewin of the Department of Chemistry, New York University, consultant to the Food and Drug Administration, was also present but was not introduced.

Dr. Schaffner asked the Johnson & Johnson group to present its findings first. Fred Pooley began by giving the results of his examination of the raw material used for "Shower-to-Shower" talcum powder. This talc is imported from a mine at Val Chisone, Italy, just north of Turin. Pooley discussed his very extensive and exhaustive tests using various techniques including megascopic identification of minerals, petrographic microscopic examination and x-ray powder diffraction study. As a result, he concluded that there was no evidence for the existence of chrysotile in any samples, even in those assembled as far back as 1949. Thus, chrysotile does not exist in the source material from which the commercial product is made.

The x-ray diffractometer work was reported independently by Professor Gordon Brown of the Department of Geology of Princeton University and by Dr. W. T. Caneer of the Colorado School of Mines Research Institute.

Both used diffractometers operated in the step-scanning method. The details of their experimental work can be described as elegant and conclusive. Neither of these competent mineralogists found any evidence for the contamination of "Shower-to-Shower" talcum with chrysotile. An outline of the work which had been done under my direction using the Guinier camera was presented next. This work had shown that, if 5% chrysotile is added to a sample of talc, the contamination is easily detectable and cannot be missed. With long exposures, as little as 1% chrysotile can be detected as a contaminant. No contaminant was found in the "Shower-to-Shower" talcum.

Walter McCrone demonstrated that chrysotile can be detected at levels as low as 1% by "optical staining" techniques. With the aid of this method, he was able to certify that "Shower-to-Shower" talc contains no chrysotile.

Jack Sheltz demonstrated the power of differential thermal analysis in detecting chrysotile. That mineral produced a record which is characterized by two specific and sharp peaks by which its presence can be determined readily down to a level of 1%. Using this test, it was evident that the Johnson & Johnson product could not contain as much as 1% chrysotile.

From these several independent investigations, it was most obvious that the Johnson & Johnson "Shower-to-Shower" talcum powder was made from a mine whose product was free from chrysotile, and that, after the product was made, it was still free from chrysotile asbestos. It must be emphasized that this common conclusion was reached by half a dozen competent scientists applying the diverse tests of x-ray diffraction by three distinct techniques, petrographic-microscopic examination by two distinct techniques, and differential thermal analysis. There can be no doubt that "Shower-to-Shower" talcum as made by Johnson & Johnson is not contaminated by any appreciable amount of chrysotile asbestos.

With this background, it was a shock to hear Professor Lewin say that, while he agreed with these results, he now relied on certain optical tests which proved that chrysotile was really present in substantial quantities in "Shower-to-Shower" talcum. He then passed out to everyone in the room a set of five sheets containing 14 sequences of three pictures to support his view that he can easily detect chrysotile in certain samples.

Professor Lewin's discourse, while beautifully delivered, contained points which cannot be accepted by trained mineralogists. His identification of chrysotile is, in my opinion, based upon an application of crystal optics which takes no account of the optics to be expected of plastically deformable layer-structures such as talc and chlorite. It also ignores crystal habit, especially the well-known habit of chrysotile.

As a type example of his identification of chrysotile, Professor Lewin showed pictures of a piece of talc, immersed in a liquid whose refractive index was 1.580, and called attention to the Becke-line test of its refractive index (a) when one edge was parallel to the vibration direction of the polarizer, and (b) when that edge was perpendicular to the vibration direction of the polarizer. The longer edge of the piece displayed a refractive index less than 1.580 when the edge was in a horizontal position on the page, whereas the rest of the piece of talc showed a higher refractive index. Since a talc crystal has two of its indices, β and γ , approximately in the plane of (001), both equal to about 1.589, the talc crystal lying on the microscopic slide might be expected to display higher refractive indices than 1.580. But, Professor Lewin pointed out, one of the edges displays a lower refractive index. Ergo: the edge is not talc but chrysotile, whose refractive indices are lower than 1.580, specifically, 1.546, 1.550 and 1.557. But this deduction overlooks the well-known plasticity of talc, the very characteristic which causes it to be soft and which recommends its use as talcum powder. Any bending of the edge of a talc crystal (and this cannot be avoided in the making of a microscopic slide, and is a characteristic which is bound to be exaggerated if its existence is ignored) results in the changing of the orientation of the edge of the talc platelet which is bent, so the lower α refractive index of

1. 539 of talc begins to be displayed. Thus, talc edges are unsafe places to determine a refractive index unless it is first ascertained that the edge in question is undamaged. This applies also to the edges of upper and lower partial flakes of a talc crystal which have become bent; such bent flakes appear to lie within the crystal. To prove that an observed low refractive index is not talc, it must be shown that it is one of the principal refractive indices of chrysotile. This Professor Lewin has not done, nor did he seem to understand this objection to his identification of the bent flake as chrysotile when it was drawn to his attention. Thus, Professor Lewin has presented absolutely no proof that any chrysotile is present in any of his samples. This negative result should be compared with all the negative tests shown by x-ray diffraction, with the competent crystal-optical studies by Walter McCrone, with the results of differential thermal analysis tests, and with the fact that there was no chrysotile in the raw material as mined, to start with.

Finally, the crystal habit of the part of the preparation which shows the low refractive index does not have the well-known habit of chrysotile asbestos. This mineral is a pyrophyllite-type sheet rolled up to form a tube with neat parallel sides and, more generally, is characterized by a cylindrical contour. None of Professor Lewin's photographed items labelled "CHRYBOTILE" conform to this habit properly. In fact, his first example is so obviously a bent-edge effect that it displays its origin by the irregular black shape of the extinguished mass when the stage is at 0° and 90°. There is another edge extinguished when the stage is at 45°.

At the end of Professor Lewin's discourse, I commented on the lack of conformity of Professor Lewin's low-index areas with the known habit of chrysotile asbestos. He attempted to dodge this objection by pointing out that the compound $Mg_6(Si_4O_{10})(OH)_8$ is polymorphous, the polymorphs being known as chrysotile, antigorite and lizardite. But, of these three, only chrysotile has the form of asbestos; the other two are layer structures and without any of the habit characteristics of asbestos (chrysotile asbestos itself has three polymorphs, α , β and γ , but all are known to have the asbestos

crystal form because all are sheet structures rolled into tubes.) Layer structures which do not have crystal habits consisting of rolled-up tubes are not more injurious to health than talc itself, for it is not the chemical composition but the asbestos-form crystal habit which is injurious.

My conclusion is that no tenable evidence has been advanced to show that any Johnson & Johnson product was made with talc which contains chrysotile asbestos, and no tenable evidence has been advanced to show that the product as sold and manufactured contains chrysotile asbestos.

Prof. G. Brown

J&J-0130698

JNJAZ55_000017718

CURRICULUM VITA: GORDON E. BROWN

Personal Data: Born Redacted - Personal Information 1943, San Diego, California;
Married. One child.

Education: B.S. Geology and Chemistry, Millsaps College, Jackson, Miss., 1965.
M.S. Mineralogy, V.P.I., 1968.
Ph.D. Mineralogy, V.P.I., 1970.

Employment Experience:

Assistant Professor, Dept. of Geological and Geophysical Sciences. Princeton University, September, 1971- present.
Post-Doctoral Research Associate, State University of New York at Stony Brook, May 1970- August 1971.
Research and Teaching Assistant, Virginia Polytechnic Institute and State University, September 1966- April 1970.
Research Assistant, Sun Oil Company, Jackson, Mississippi, June 1963- September 1965.

Professional Societies:

Mineralogical Society of America
American Geophysical Union
American Association for the Advancement of Science

Research Experience:

G.E. Brown investigated the crystal chemistry of the olivines as part of his doctoral research. The crystal structures of Ni-, Co-, (Fe,Mn)- (Mn,Mg)- (Fe,Ca)- and (Mn,Ca)- olivines were refined from single crystal X-ray data and cation ordering was studied in the (Fe,Mn)- and (Mn,Mg)- olivines. Earlier efforts were devoted to a study of bonding in the tetrahedral portions of silicates in collaboration with G.V. Gibbs. During post-doctoral work at Stony Brook, he collaborated with C.T. Prewitt and J.J. Papike on X-ray studies of the Apollo 12 clinopyroxenes and olivines. Included in this work are high-temperature X-ray studies of the $P2_1/c \rightleftharpoons C2/c$ transition in intergrown lunar pigeonite/angites, design of several high temperature furnaces for single crystal X-ray work (also in collaboration with S. Sueno), high temperature structural studies of pigeonite and cummingtonite, order-disorder studies of lunar olivines over a range of temperatures, and a neutron diffraction study of Al/Si ordering in sanidine.

Publications - G.E. Brown

- G.E. Brown and G.V. Gibbs (1968) The crystal structure of osumilite (Abstract) Geol. Soc. Amer. S.E. Sec. Meet., Durnham, N.C., 89.
- G.E. Brown and G.V. Gibbs (1969) Refinement of the crystal structure of osumilite. Amer. Mineral. 54, 101-116.
- G.E. Brown, G.V. Gibbs and P.H. Ribbe (1969) The role of d-p π -bonding in minerals containing SiO_4^{4-} , AlO_4^{5-} and MgO_4^{6-} tetrahedral ions (Abstract) Geol. Soc. Amer. S.E. Sec. Meet., Columbia, S.C., 11.
- G.E. Brown, G.V. Gibbs and P.H. Ribbe (1969) The nature and variation in length of the Si-O and Al-O bonds in framework silicates (Abstract). Trans. Amer. Geophys. Union 50, 358.
- G.E. Brown, G.V. Gibbs and P.H. Ribbe (1969) The nature and variation in length of the Si-O and Al-O bonds in framework silicates. Amer. Mineral. 54, 1044-1061.
- G.E. Brown and G.V. Gibbs (1969) Oxygen coordination and the Si-O bond. Amer. Mineral. 54, 1528-1539.
- G.E. Brown and G.V. Gibbs (1970) The variation of the Si-O bond lengths, Si-O-Si and O-Si-O angles in the chain and sorosilicates. (Abstract) Amphibole-Pyroxene Symposium, Blacksburg, Va. Amer. Mineral. 55, 298.
- G.E. Brown and G.V. Gibbs (1970) Stereochemistry and ordering in the tetrahedral portion of silicates. Amer. Mineral. 55, 1587-1607.
- G.E. Brown, G.A. Novak, K.G. Robinson, G.V. Gibbs and P.H. Ribbe (1970) Crystal chemistry of the orthosilicates (Abstract) Geol. Soc. Amer. Annual Meeting, Milwaukee. 2(7), 504.
- J.J. Papike, A.E. Bence, C.T. Prewitt and G.E. Brown (1970) Apollo 12 clinopyroxenes: subsolidus relations (Abstract) Geol. Soc. Amer. Annual Meeting, Milwaukee. 2(7), 644.
- A.E. Bence, G.E. Brown, J.J. Papike, C.T. Prewitt and C.H. Wu (1971) Apollo 12 clinopyroxenes: chemical trends, epitaxy, exsolution and phase transitions. (Abstract) Proc. Apollo 12 Lunar Sci. Conf., Houston, Texas. 105-106.

J&J-0130700

- J.J. Papike, A.E. Bence, G.E. Brown, C.T. Prewitt and C.H. Wu (1971) Apollo 12 clinopyroxenes: exsolution and epitaxy. *Earth Planet. Sci. Lett.* 10, 307-315.
- C.T. Prewitt, G.E. Brown and J.J. Papike (1971) Apollo 12 clinopyroxenes: high temperature X-ray diffraction studies. *Proceedings 2nd Lunar Science Conference.* 1, 59-68.
- G.E. Brown, C.T. Prewitt and J.J. Papike (1971) Structural studies of intergrown lunar pyroxenes (Abstract) *Trans. Amer. Geophys. Union* 52, 270.
- G.E. Brown, W.C. Hamilton, C.T. Prewitt and S. Sueno (1971) Neutron diffraction study of Al/Si ordering in sanidine (Abstract) *Geol. Soc. Amer. Annual Meeting, Washington.* 3(7), 514.
- G.E. Brown, C.T. Prewitt, J.J. Papike and S. Sueno (1972) A comparison of the structures of low and high pigeonite. *Jour. Geophys. Res.* (in press).
- S. Sueno, J.J. Papike, C.T. Prewitt and G.E. Brown (1972) The crystal structure of high-cumingtonite. *Jour. Geophys. Res.* (in press).

X-ray Diffraction Study of Johnson & Johnson's Shower-to-Shower
Talcum Powder

Gordon E. Brown

Assistant Professor of Mineralogy and Crystallography
Princeton University
Princeton, New Jersey

Abstract

An x-ray diffraction study of Lewin's sample of Johnson & Johnson's Shower-to-Shower talcum powder using slow, continuous scanning, diffractometer techniques with monochromatized Cu radiation showed that two forms of chlorite, phlogopite, magnesite, dolomite and calcite are minor contaminants (<10% total) of the talc. No evidence of chrysotile or tremolite in Lewin's sample was found. Examination of the 2θ regions around 12° and 24° alone is not necessarily sufficient to establish the presence of chrysotile asbestos because of the similar peak positions of two other non-fibrous serpentine polymorphs, lizardite and antigorite. However, detailed examination of the range $5-80^\circ$ 2θ does permit discrimination between chlorite and serpentine peaks, assuming that these minerals are present in detectable amounts.

The x-ray data on J&J's Shower-to-Shower powder presented by S.Z. Lewin in a written report (May 16, 1972) to the Cosmetic, Toiletry and Fragrance Association and during a hearing (Sept. 21, 1972) before officials of the Food and Drug Administration show no evidence of chrysotile or tremolite when examined carefully. Moreover, the fibrous-looking particles in the photomicrographs also presented by Lewin at the Sept. 21 hearing can best be interpreted as rolled talc particles, which are sometimes produced during grinding. Lewin's claim that chrysotile is present in J&J's Shower-to-Shower powder is not supported by his data or by any of the results (from x-ray and electron diffraction, optical dispersion staining, transmission and scanning electron microscopy, electron microprobe analysis, differential thermal analysis, petrographic examination of the uncrushed Italian talc and detailed field study of the Italian mine) amassed by Johnson and Johnson's team of consultants.

J&J-0130702

Introduction

Talc deposits can result from a variety of processes including hydrothermal alteration of ultrabasic rocks (steatitization) and thermal metamorphism of siliceous dolomites (Turner, 1948). In the former process serpentinization may or may not occur before the development of talc. In either case the resulting talc may contain minor contaminants which can occur from incomplete steatitization, retrograde metamorphic processes or more commonly simultaneous crystallization of additional phases necessary to maintain mass balance. Contaminants such as magnesite, dolomite, calcite, chlorite, serpentine, tremolite, actinolite, phlogopite and quartz may result.

It is the purpose of this study to identify the contaminants present in Johnson & Johnson's Shower-to-Shower talcum powder using x-ray diffraction techniques. Because of S.Z. Lewin's claim that this powder contains ~ 5% chrysotile by weight, particular care was taken to verify or reject its presence using a continuous scanning x-ray technique similar to that employed by Lewin. The sensitivity of the continuous scanning technique to small concentrations of chrysotile in talc is estimated to be detection of ~2-3% chrysotile by weight. Lewin in a report to CTFA (5-16-72), claims a sensitivity of 1-2% chrysotile using a scanning rate of $1^\circ 2\theta/\text{min}$. Therefore, if chrysotile is present in the Shower-to-Shower powder in amounts of ~5% by weight, as claimed by Lewin, its presence should be easily detectable.

Experimental

A powder mount of Lewin's sample of J & J's Shower-to-Shower powder was prepared by mixing 0.1 gr. of powder (< 325 mesh) with a mixture of acetone and duco cement on a glass slide. A very small quantity (~0.01 gr.) of powdered Si metal (325 mesh), for use as an internal standard, was sprinkled onto this mixture and stirred in. Powder mounts of three chemically diverse chrysotiles were also prepared in the same manner.

All x-ray data were collected using a Norelco vertical diffractometer mounted on a Norelco generator with a Cu standard focus x-ray tube. The diffractometer is equipped with a sample spinner, which is used to reduce the effects of preferred orientation, and a LiF crystal monochromator, which is set to produce a beam of Cu $K\alpha$ x-rays. The PHA was tuned using the 111 peak of Si-metal and 2 V baseline and window settings were found satisfactory. Diffractometer alignment was then checked using Si-metal peaks which were located within $0.01^\circ 2\theta$ of their correct values. 1° source and receiving slits were used in all runs. Smaller receiving slits, which are used to increase resolution, were found

to reduce intensity significantly. Enhancement of intensity was considered more important than increasing resolution because of the small amounts of impurities being searched for. All tracings were run at 40 KV and 20 mA using a scan speed of $1/4^\circ$ 2θ /min. and a time constant =2. Scans from $5-80^\circ$ 2θ were run for the Shower-to-Shower sample and the three crysotiles. Table 1 contains the 2θ values and d-spacings for all peaks in this interval for the Shower-to-Shower sample and Table 2 lists the chemical compositions and positions of the three strongest peaks for the three crysotile specimens studied. Figure 1 is a copy of the diffractogram for the Shower-to-Shower sample.

Interpretation

The interpretation of a diffraction pattern from a complex mixture of phases is always difficult and necessitates great care. The standard philosophy employed in such an interpretation is outlined in Klug and Alexander (1954) and simply requires that all peaks be explained using the ASTM Powder Diffraction File or other standard patterns. The patterns in the ASTM file are for minerals of specific compositions, so the effects of solid solution on d-spacings must be taken into account. Because of the paragenesis of talc, a number of specific contaminants are expected and were searched for. In addition to peaks due to talc and the Si internal standard, 22 extra peaks were recorded in the diffraction pattern of J & J's Shower-to-Shower powder (See Table 1 and Figure 1). These additional peaks are due to two varieties of chlorite ($\sim 5\%$ by weight), phlogopite ($\sim 2\%$), magnesite ($\sim 2\%$), dolomite ($\sim 1\%$) and calcite (trace). Chlorite has the hypothetical composition $Mg_{12}Si_8O_{20}(OH)_{16}$, however, Al can substitute for Mg and Si. When Al replaces about 3 Si per formula unit, the chlorite is called sheridanite; when about 1 Al substitutes for Si, it is termed a penninite. Because tetrahedral Al and Si ions differ by about $0.14\text{\AA}$ in size, penninite and sheridanite have different d-spacings ($\Delta \sim 0.1\text{\AA}$) and 2θ positions. The main peak at $7.15\text{\AA}$ and its low angle shoulder are therefore interpreted to be due to sheridanite and penninite, respectively, with sheridanite and penninite constituting $\sim 4\%$ and $\sim 1\%$ by weight of the sample. For each of the contaminants listed above, identification was not accepted unless their peaks showed self-consistent intensity relationships. The presence of tremolite was not confirmed because its three strongest peaks (8.38 , 3.12 and $2.70\text{\AA}$) are absent. Likewise, chrysotile can be rejected as a component because its two strongest peaks, the 002 and 004, predicted to occur at 7.31 and $3.65\text{\AA}$, respectively, are missing. Although the low angle shoulder ($\sim 7.2\text{\AA}$) to the 002 chlorite peak ($7.15\text{\AA}$) overlaps the $7.3\text{\AA}$ region, it is due to the 002 of penninite, not the 002 of crysotile. If the 002 of crysotile were present,

the 004 of chrysotile is required to be present at 3.65Å and should have nearly the same relative intensity as the shoulder at 7.2Å. Examination of Figure 1 shows no peak in the 3.65Å region. Preferred orientation, which undoubtedly would have occurred for chrysotile if present, would enhance all of its basal reflections, not just the 002. One might argue that substitution of Al and Fe for Si and Mg in chrysotile would cause a shift in d-spacings such that the 002 and 004 of chrysotile are coincident with the chlorite peaks at 7.22-7.15Å and 3.56Å. To test this hypothesis, diffraction patterns of three chemically diverse chrysotiles were run and the results are reported in Table 2. The effects of these chemical substitutions on the basal spacing of chrysotile appear to be small. Therefore, it is doubtful that the 004 chlorite peak at 3.65Å obscures an 004 of chrysotile.

A word of caution is necessary even if peaks occur at 7.31 and 3.65Å. Besides chrysotile, there are two other serpentine polymorphs known as lizardite and antigorite which differ slightly in structure and totally in morphology (both are platy) from chrysotile. However, the two strongest peaks of each polymorph occur near 7.36 and 3.66Å (Whittaker and Zussman, 1956; Page and Coleman, 1967). Whittaker and Zussman (1956) emphasize that correct identification of a serpentine polymorph is possible only if a complete and accurate diffraction pattern is obtained. Obviously discrimination of this type would be impossible if small amounts of one or more of the serpentine polymorphs did occur in the Shower-to-Shower powder because of the weakness of the serpentine pattern and possible interference from other peaks.

Conclusions

The 22 extra peaks observed in the x-ray pattern of Lewin's sample of J&J's Shower-to-Shower talcum powder are interpreted as being due to sheridanite (4%), penninite (1%), phlogopite (2%), magnesite (2%), dolomite (1%) and calcite (trace). No evidence for chrysotile or tremolite was found. The diffraction patterns of three chemically different chrysotiles were run to check the effects of cation substitutions on basal spacings. These effects were found to be minor and it is concluded that the 004 peak of chlorite does not obscure an 004 peak of chrysotile. Although the x-ray diffraction pattern from a multi-component mixture such as Shower-to-Shower is complicated, it can be interpreted by careful use of the ASTM Powder Diffraction File.

Gordon E. Brown

J&J-0130705

JNJAZ55_000017725

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Table 1: X-ray Data for Lewin's Sample of Shower-to-Shower Powder

hkl	Mineral	I/I ₀ ***	d(A)	2θ(deg.)
001	Chlorite	60	14.5-14.3	6.10-6.15
001	Phlogopite	100	9.9-9.8	8.90-9.00
002	Talc	100	9.47	9.34
002	Chlorite(P)**	100	7.22	12.26
002	Chlorite(S)**	70	7.15	12.38
003	Chlorite(S+P)	50	4.75	18.70
004	Talc	90	4.70	18.88
020,111	Talc	30	4.59	19.35
020	Chlorite(S+P)	40B	4.58	19.39
004	Chlorite(S+P)	50(S) or 100(P)	3.56	25.00
022	Phlogopite	20	3.37	26.42
003	Phlogopite	100	3.35	26.60
111	Si Standard	100	3.14	28.46
006	Talc	100	3.13	28.65
220	Calcite	100	3.02	29.50
104	Dolomite	100	2.90	30.87
005	Chlorite(P)	60	2.88	31.02
005	Chlorite(S)	20	2.85	31.42
104	Magnesite	100	2.75	32.54
200,131	Phlogopite	30	2.63	33.98
132	Talc	30	2.61	34.42
131,202	Chlorite(S+P)	40	2.59	34.60
132,204	Talc	65	2.48	36.18
132,203	Chlorite(S+P)	50	2.44	36.8
008	Talc	16*	2.34	38.42
134	Talc	20	2.21	40.75
136	Talc	20	2.11	42.85
113	Magnesite	43	2.10	
135,204	Chlorite(S+P)	40	2.02	44.85
220	Si-Standard	60	1.92	47.18(K _{α1})
	Si-Standard		1.92	47.33(K _{α2})
0-0-10	Talc	40*	1.87	48.56
116	Magnesite	34	1.70	
244,138	Talc	20B	1.68	54.72°
311	Si-Standard	35	1.64	56.02(K _{α1})
0-0-12	Talc	20*	1.56	59.16
331,060	Chlorite(S+P)	20	1.54	60.32
060,332	Talc	40	1.53	60.55(K _α)
1-3-10	Talc	8B	1.46	63.2
139,208	Chlorite(P)	30	1.41	66.2
2-0-10	Talc	16	1.41	67.0
1-3-12	Talc	20	1.39	67.1
400	Si-Standard	8	1.36	69.20

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620

<u>hkl</u>	<u>Mineral</u>	<u>I/Io</u>	<u>d (Å)</u>	<u>2θ (deg.)</u>
0·0·14	Talc	16*	1.34	70.34
264	Talc	108	1.30	72.90
331	Si-Standard	13	1.25	76.30

*Enhanced by preferred orientation

** (S)= Sheridanite; (P)= Penninite

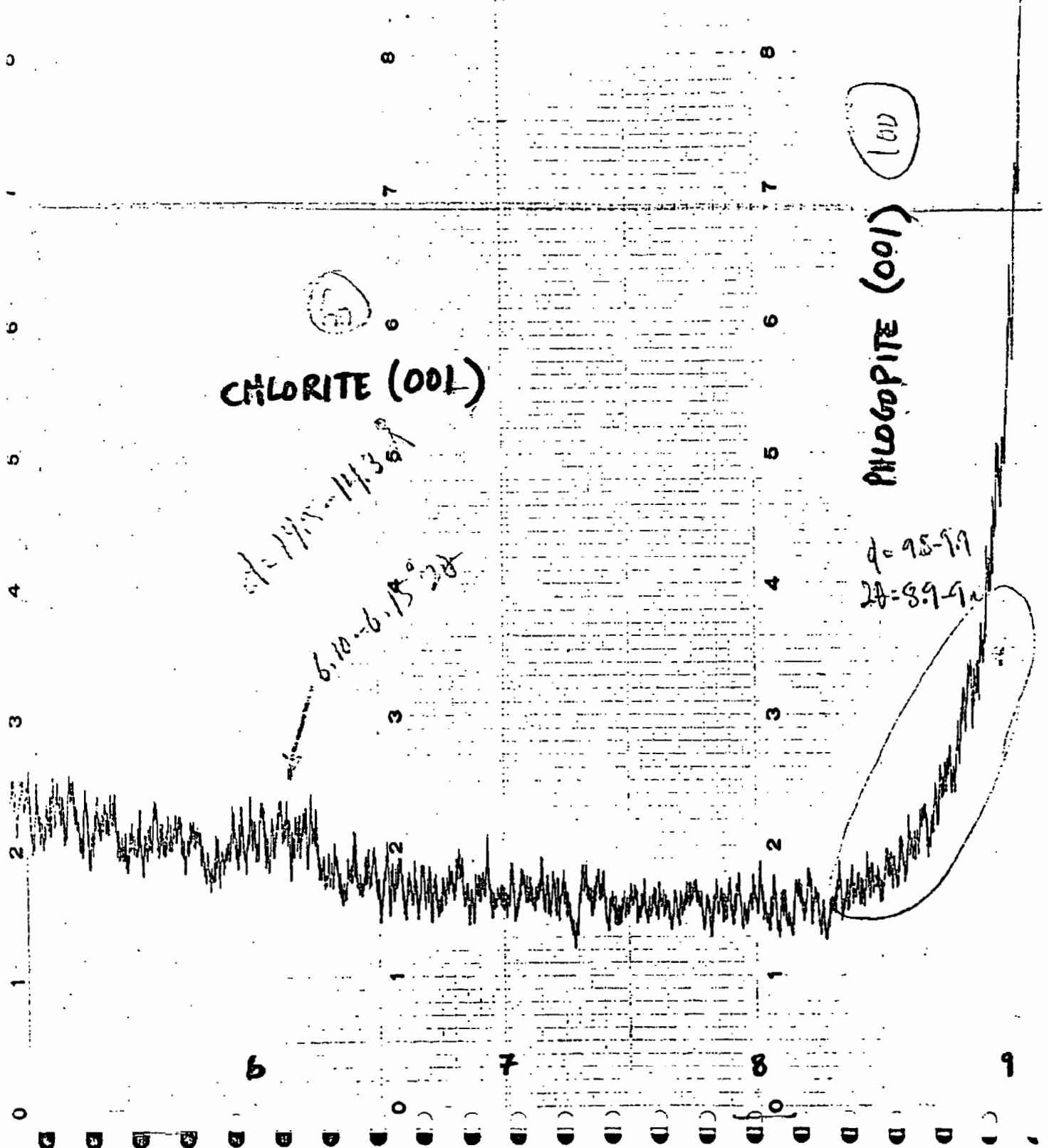
*** ASTM Intensity

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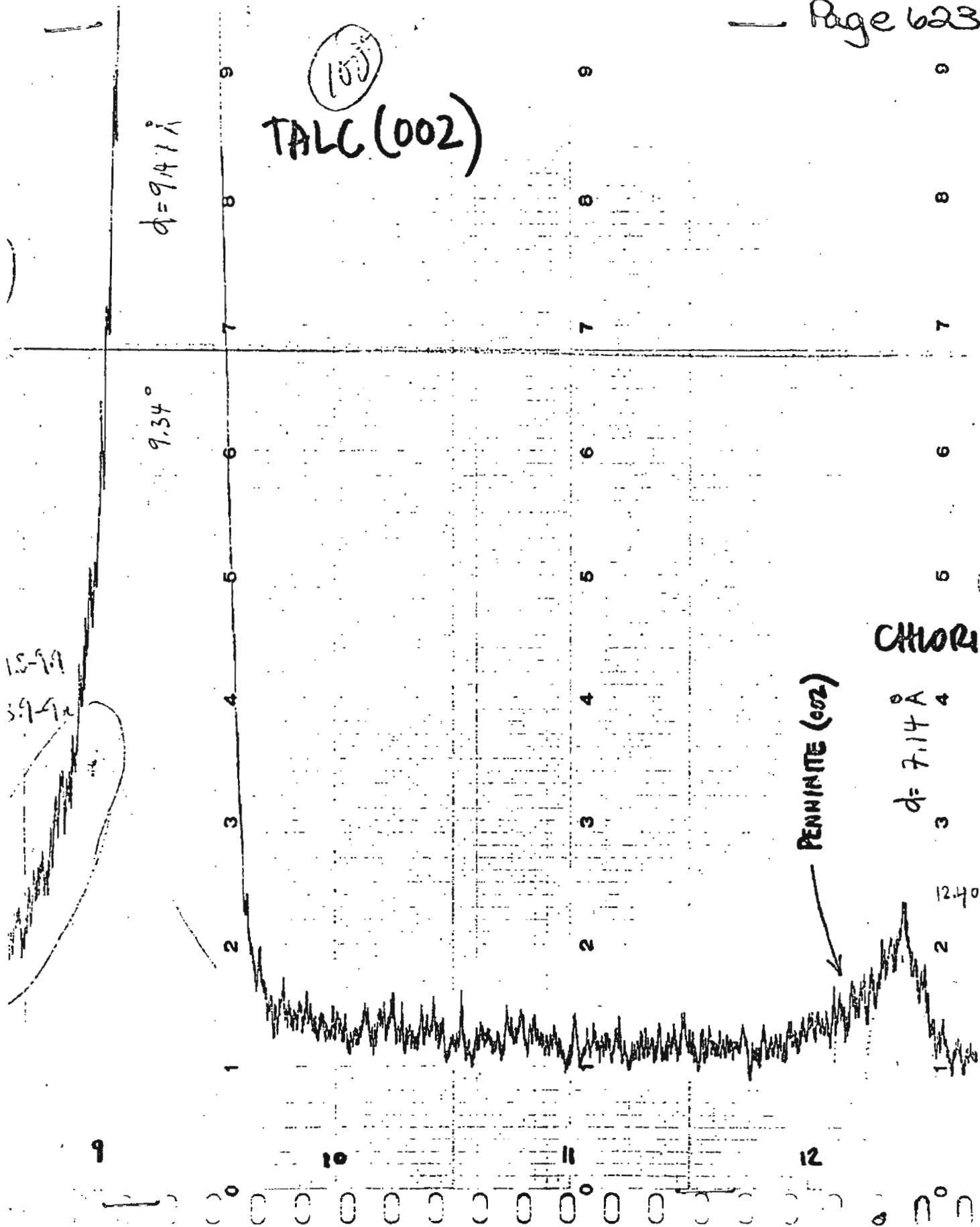
Table 2: Chemical and X-ray Data for Chrysotile

	<u>Jeffrey</u>	<u>Arizona</u>	<u>Coalinga</u>
Tetrahedral Cations	Si _{3.91} Al _{0.07}	Si _{3.98} Al _{0.02}	Si _{3.82} Al _{0.18}
Octahedral Cations	Mg _{5.78} Fe _{0.19}	Mg _{5.93} Fe _{0.05}	Mg _{5.62} Al _{0.41} Fe _{0.15}
d ₀₀₂ (Å)	7.36	7.35	7.31
d ₀₀₄ (Å)	3.66	3.66	3.65
d ₀₆₀ (Å)	1.54	1.54	1.54

FIGURE 1: X-RAY PATTERN OF LENIN'S SAMPLE
OF J & J'S SHOWER-TO-SHOWER POWDER

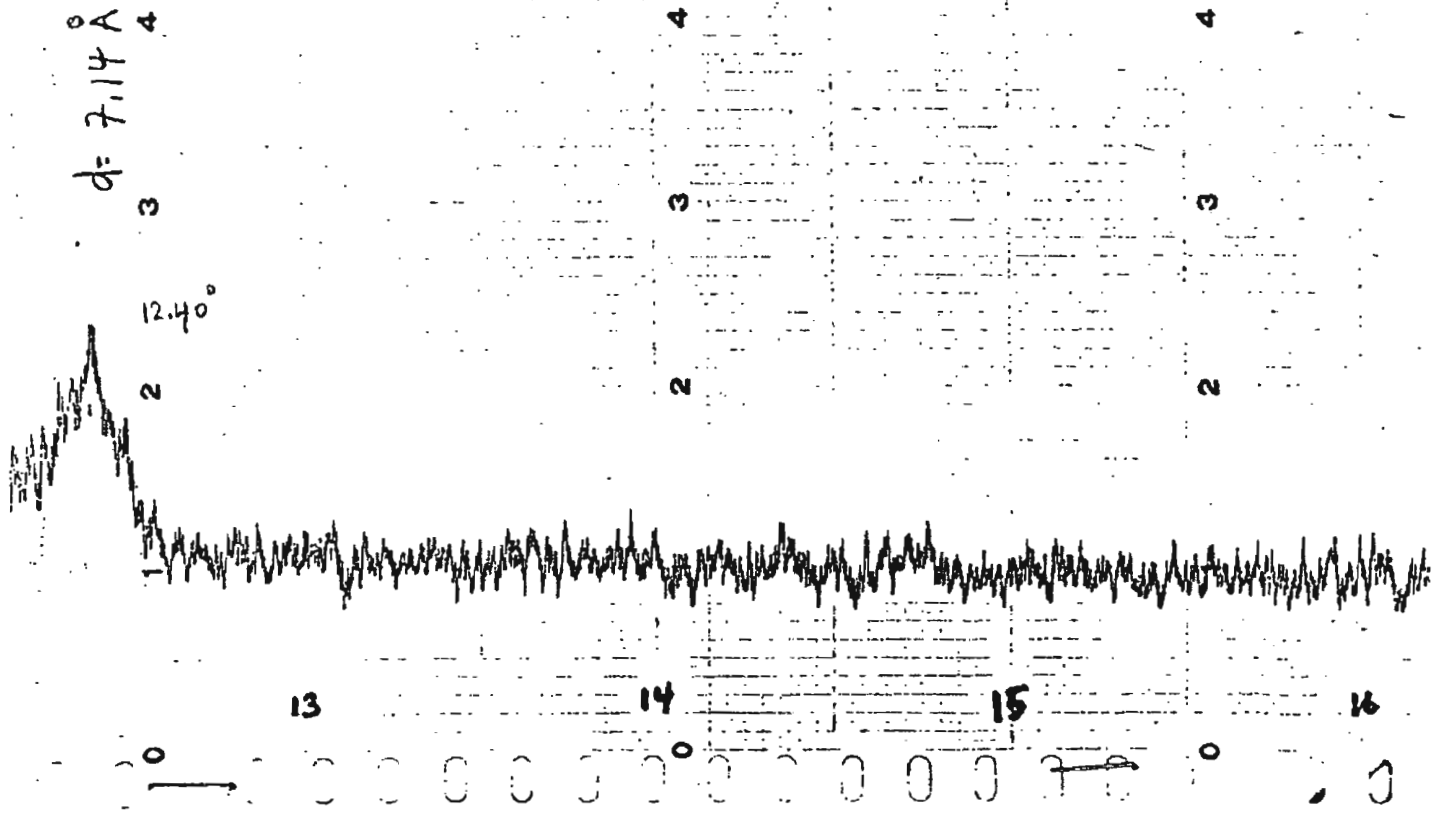


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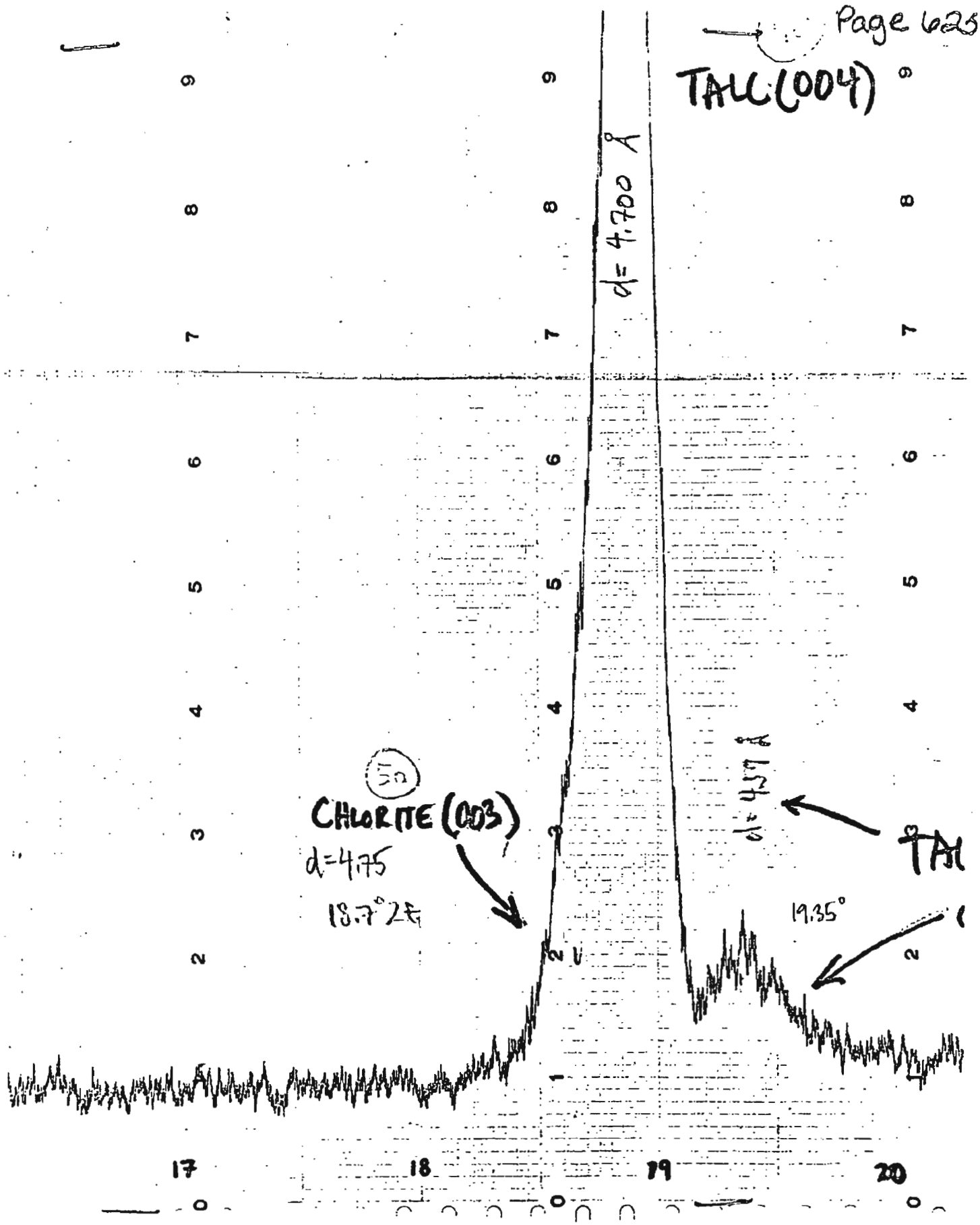


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CHLORITE (002)



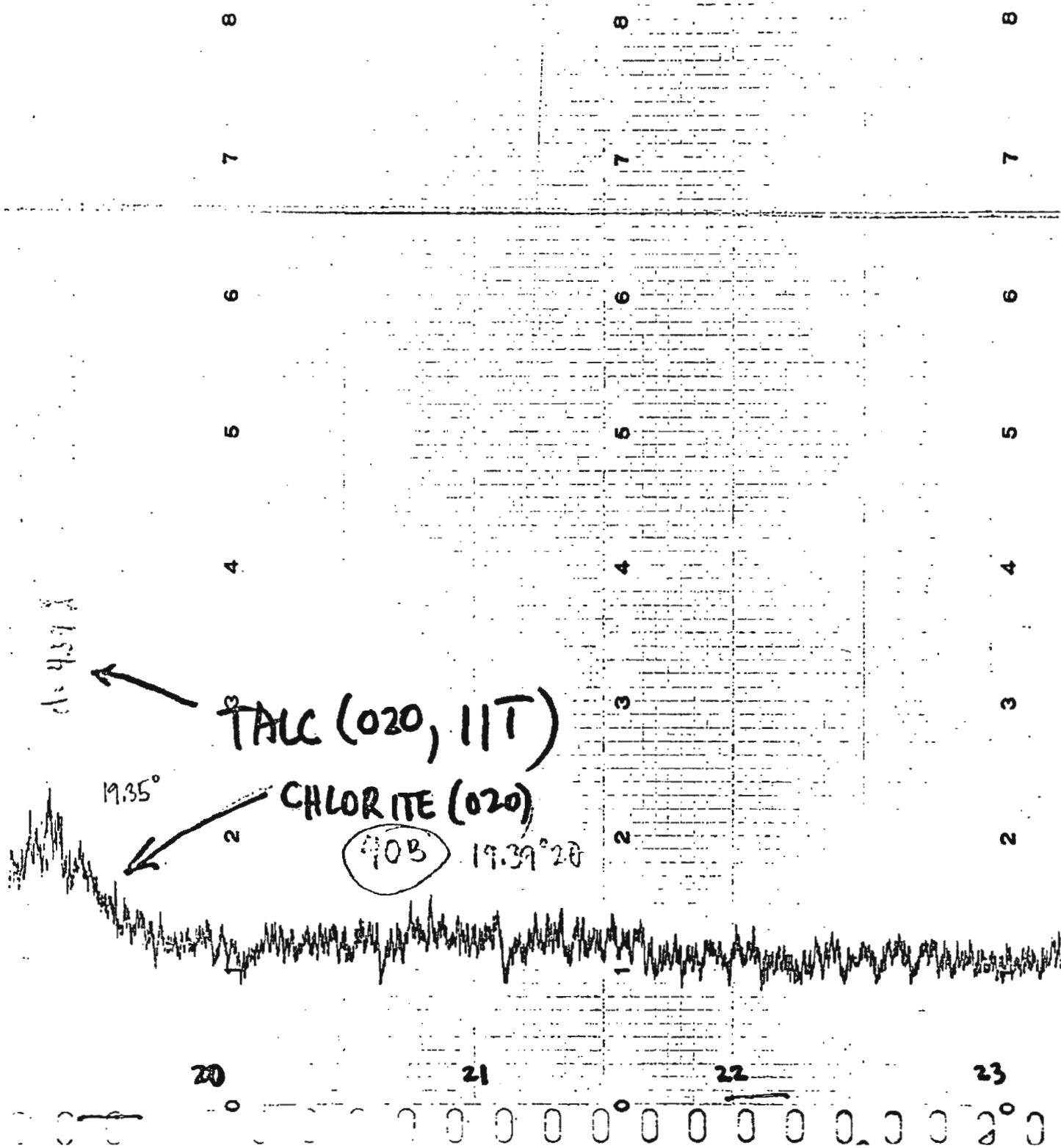
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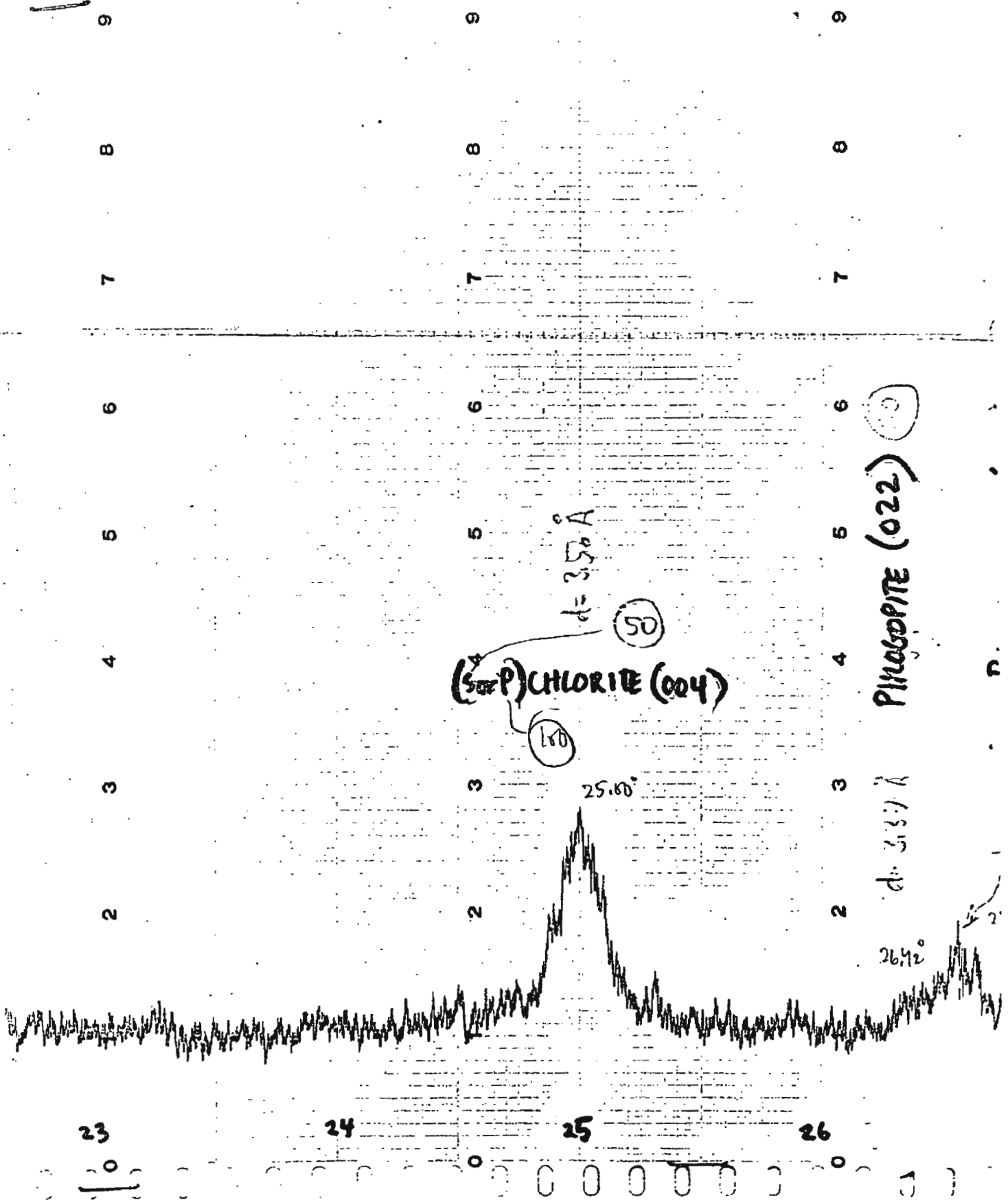
Page 625
 TALC (004) °

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ALC(004)

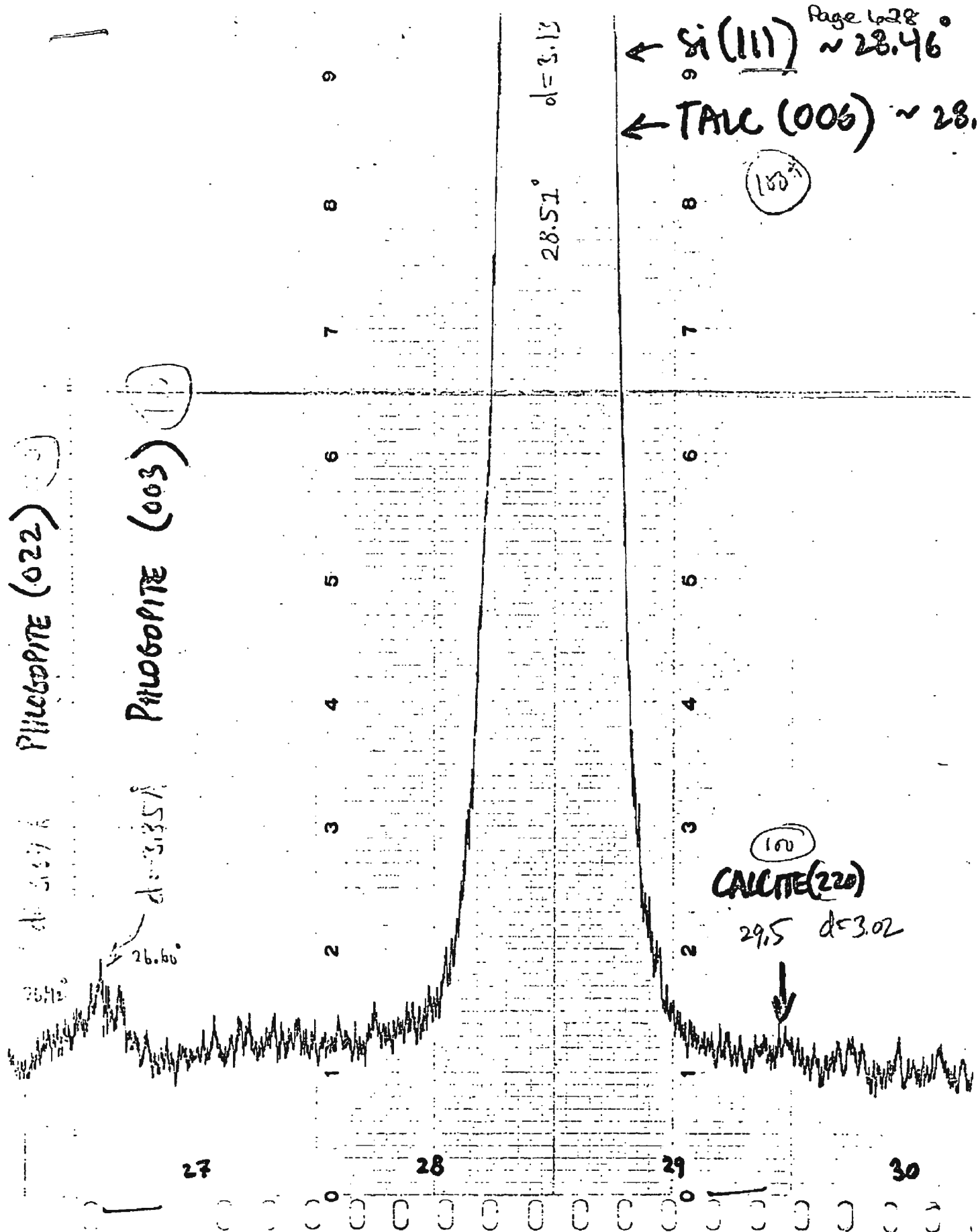


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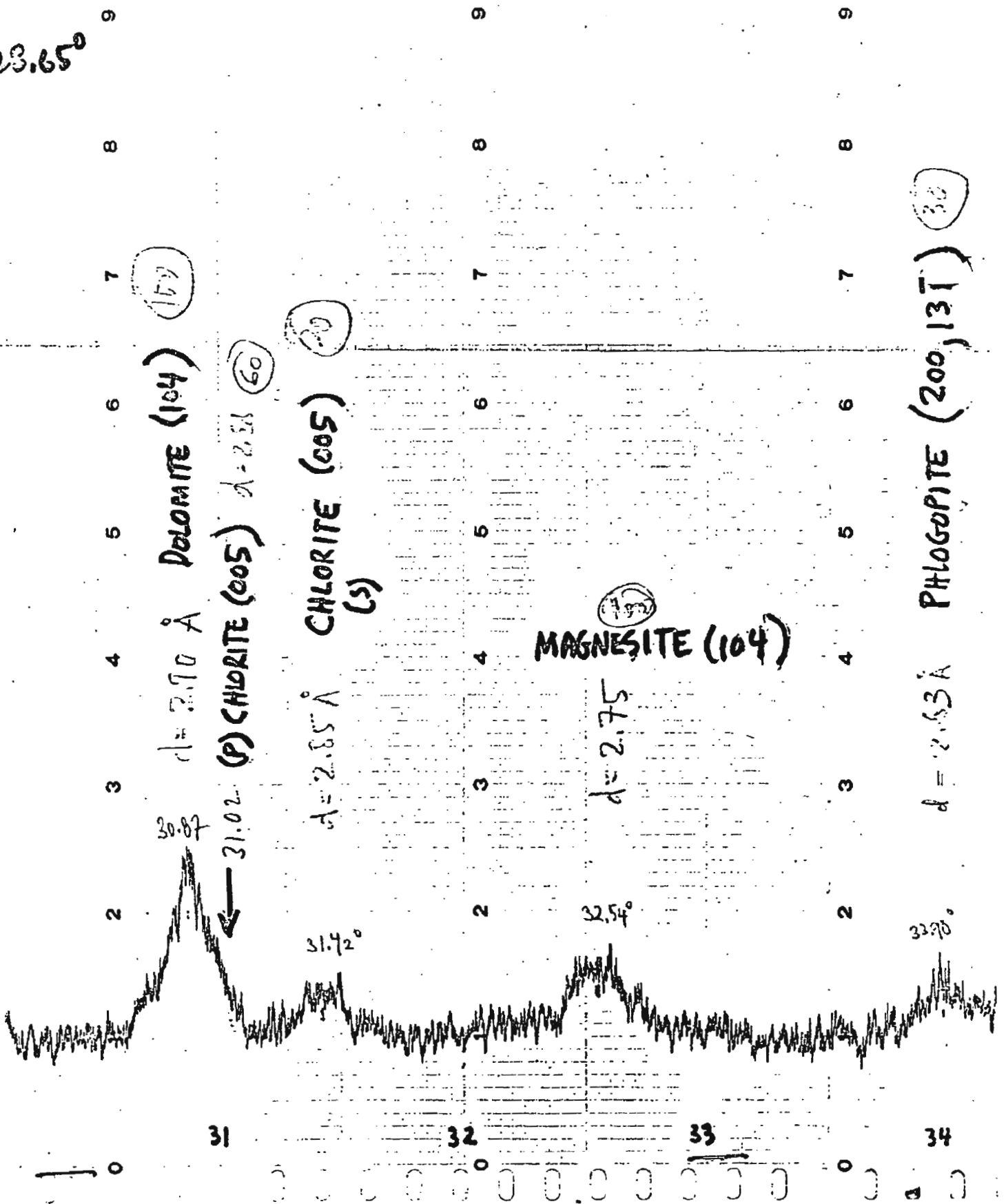
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28.65°



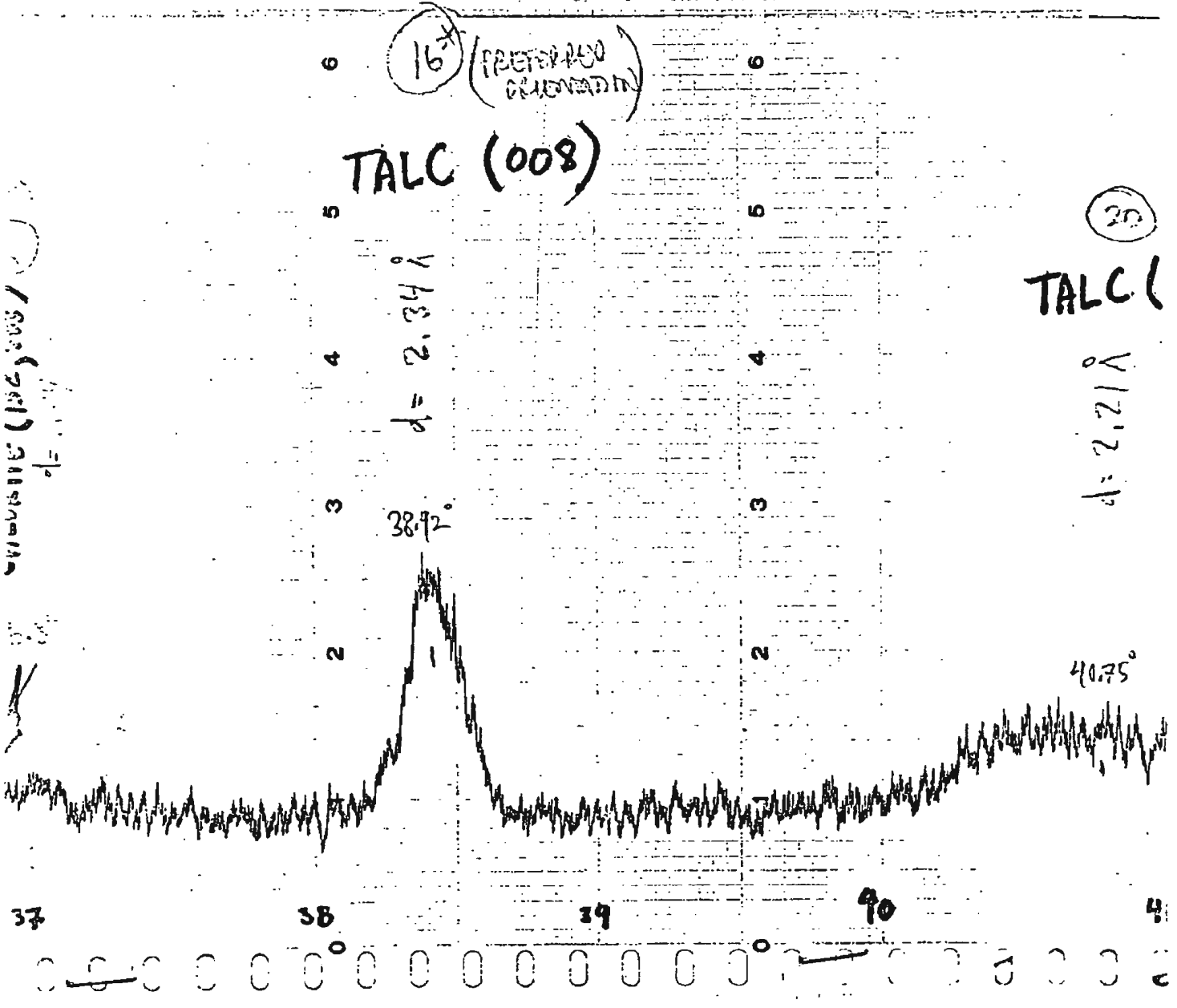
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(20)
TALC (134)

$d = 2.21 \text{ \AA}$

(20)
TALC (136)

$d = 2.11 \text{ \AA}$

MAGNESITE (113)
2.102

COULD MAKE
UP FROM IF
THIS PEEK

40.75°

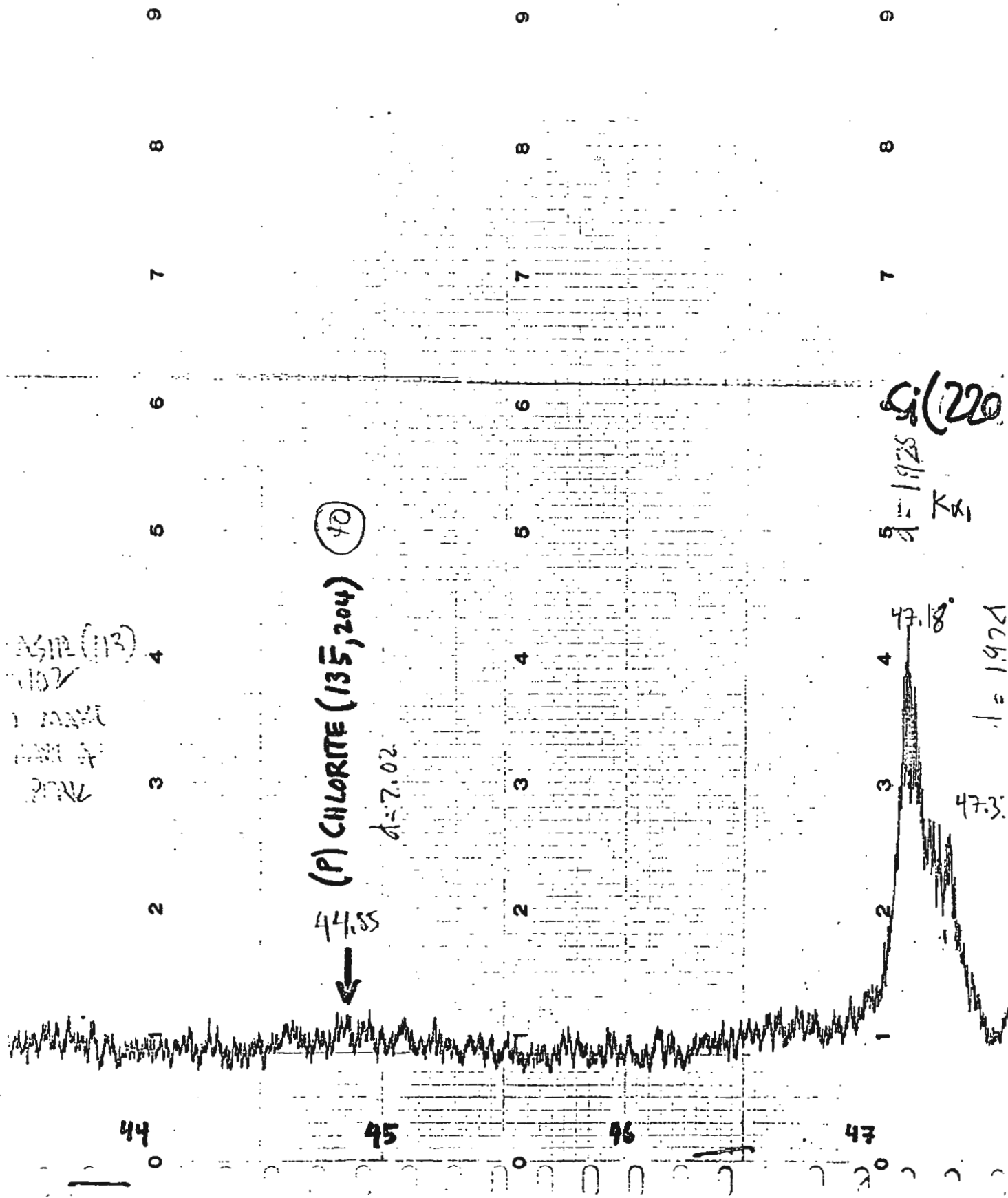
42.85°

41

42

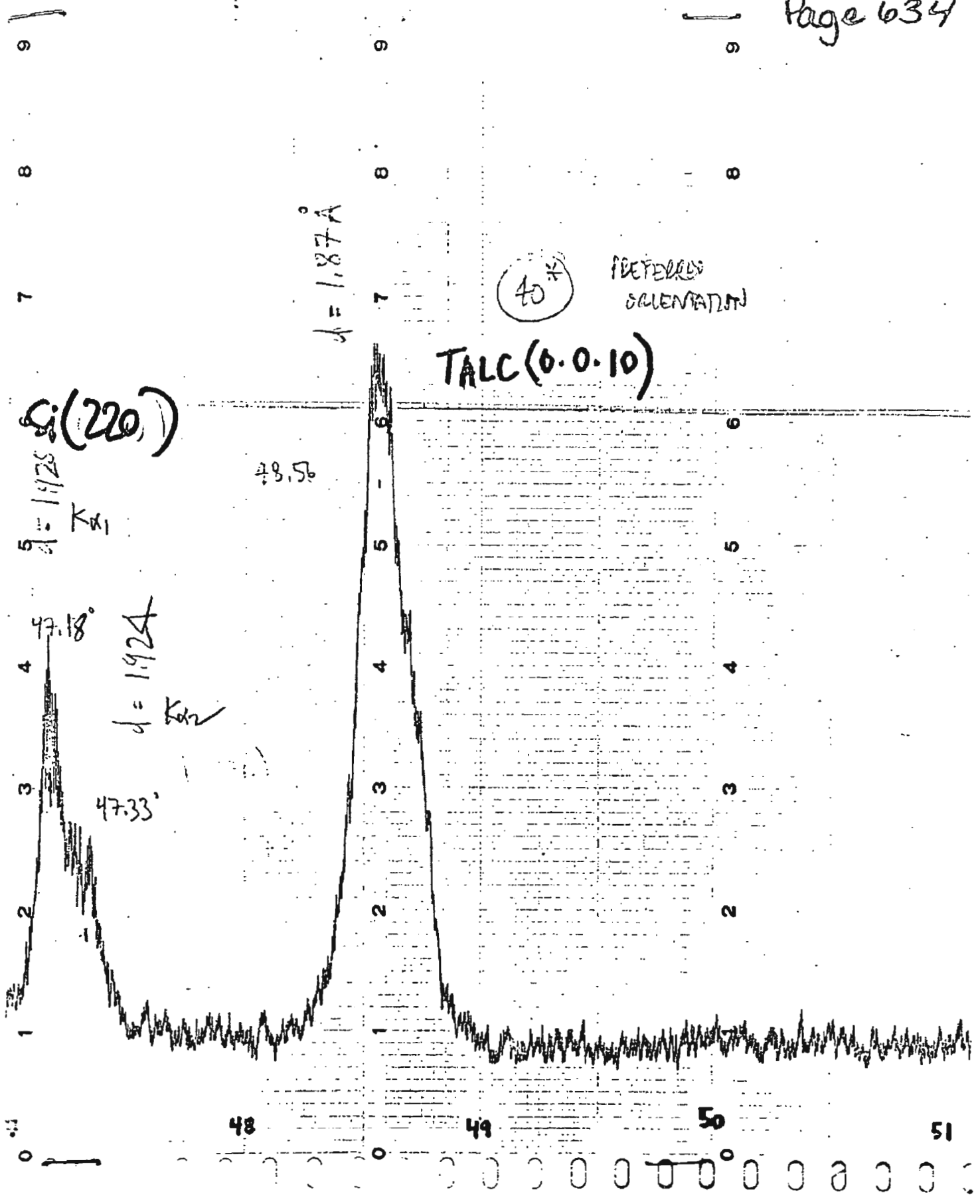
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44



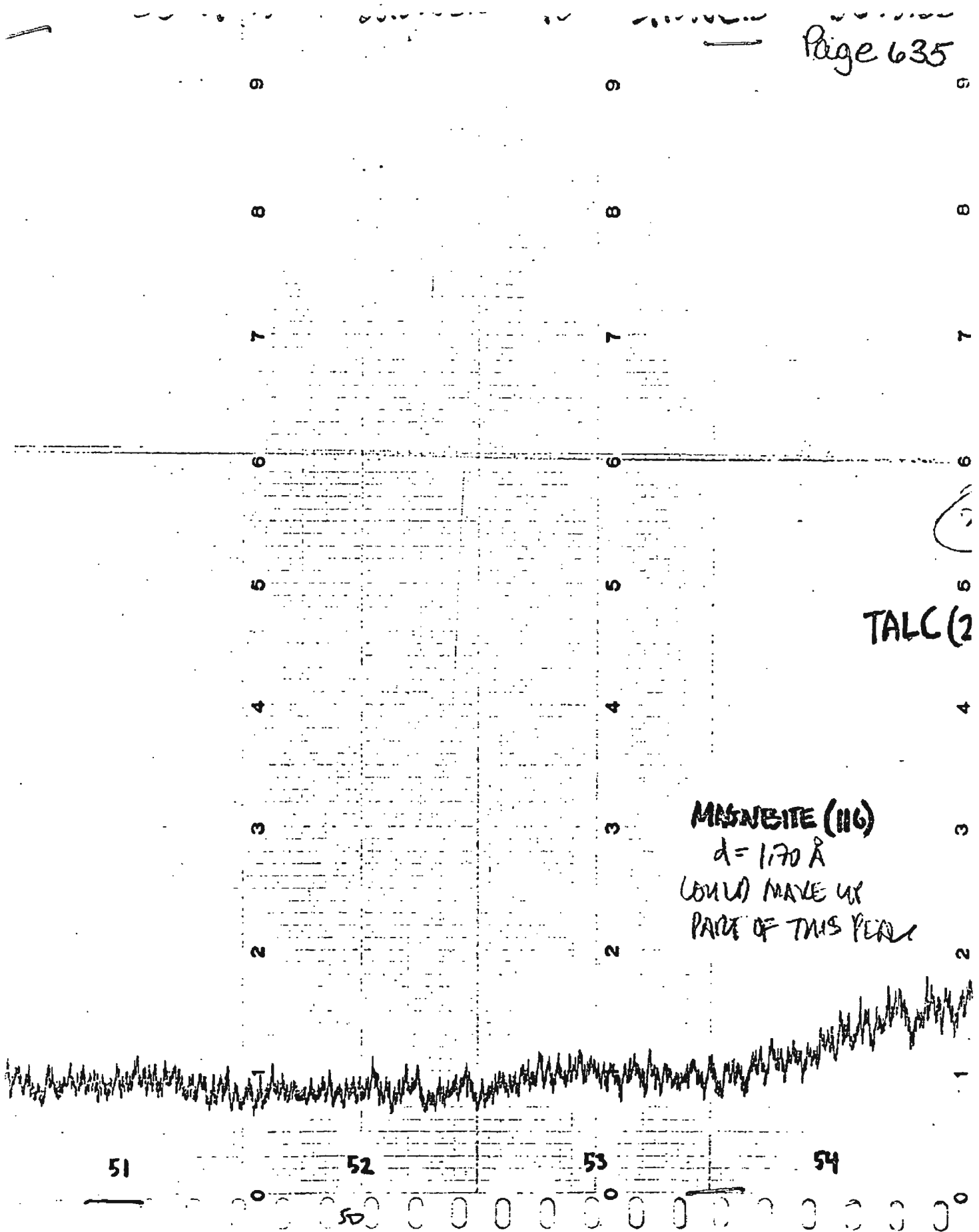
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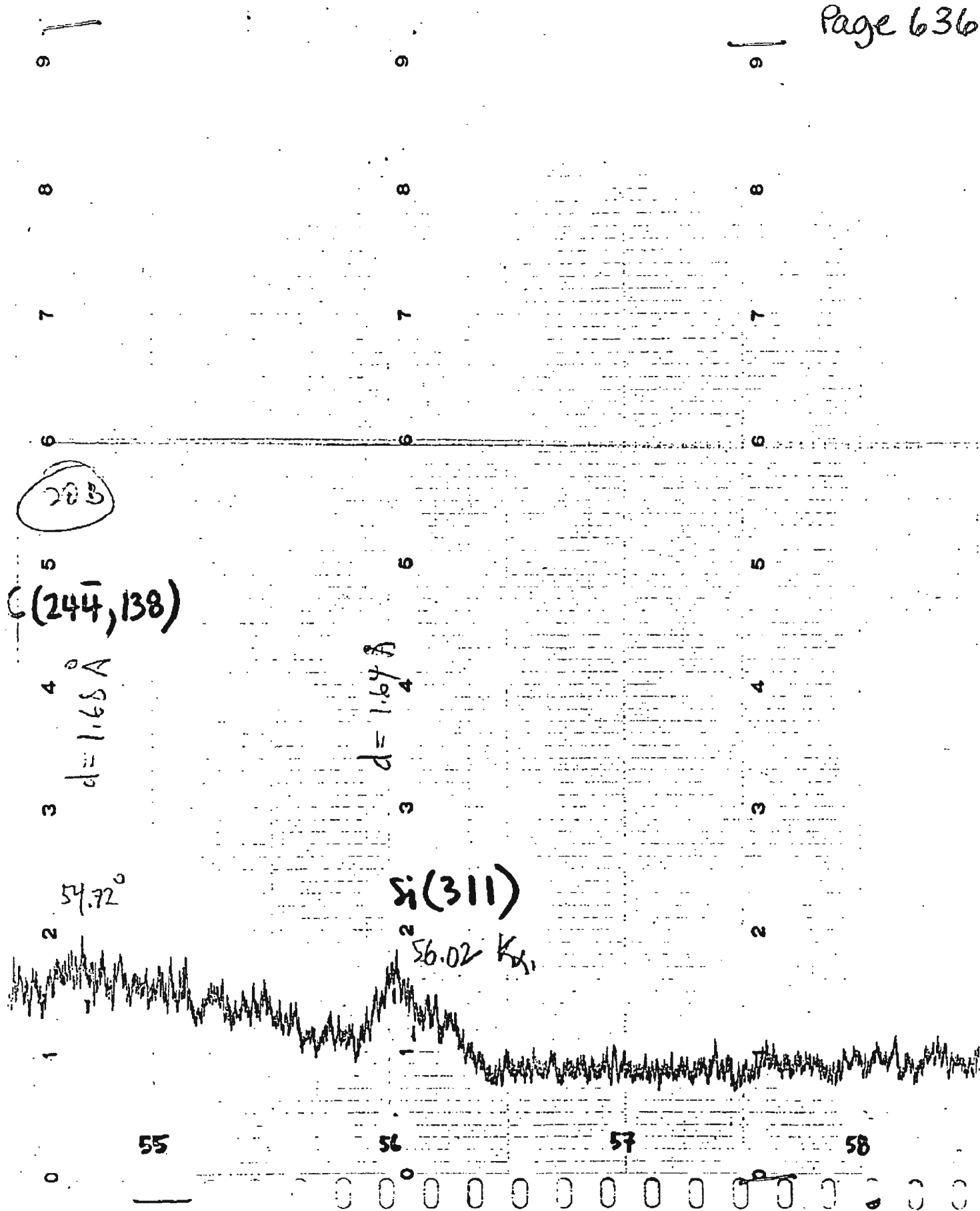
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PRETEXTED
ORIENTATION

(25)

TALC (0.0.12)

$d = 1.56 \text{ \AA}$

59.16

(20)
CHLORITE (P.S.)
(331, 060)
 $d = 1.54$

(40)

TALC (060, 332)

$d = 1.53 \text{ \AA}$

60.55

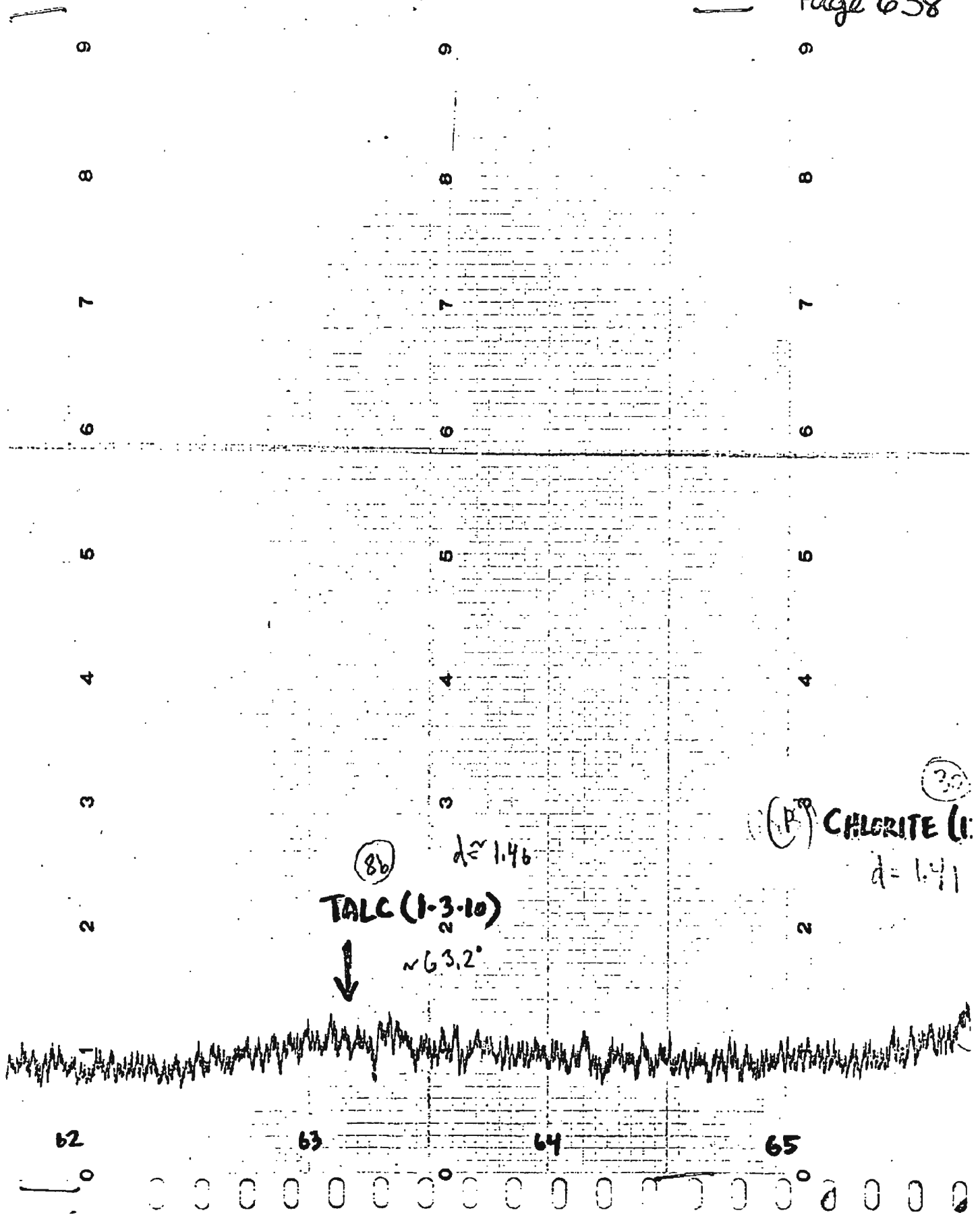
$K_{\alpha 1}$ $K_{\alpha 2}$

59

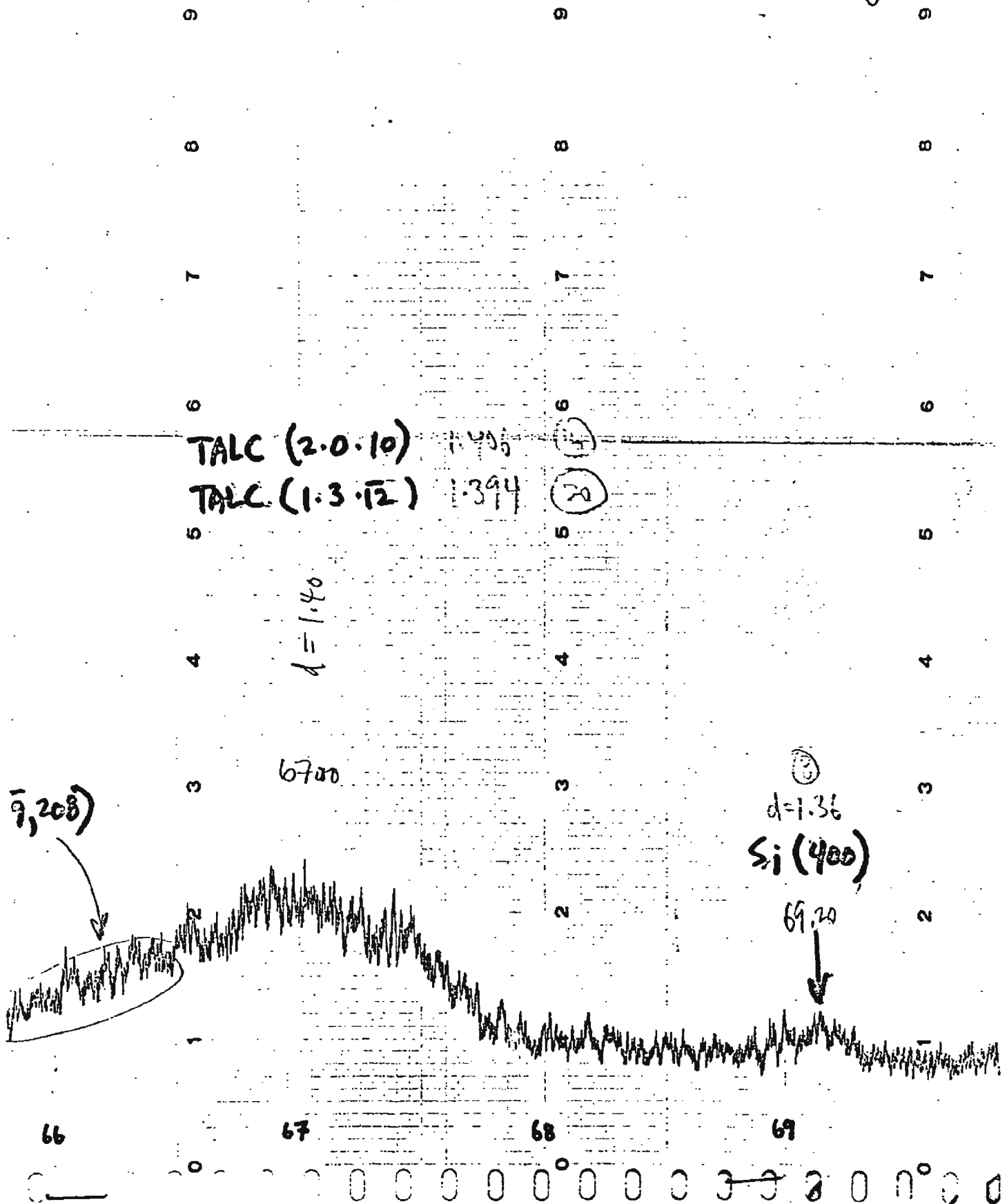
60

61

62

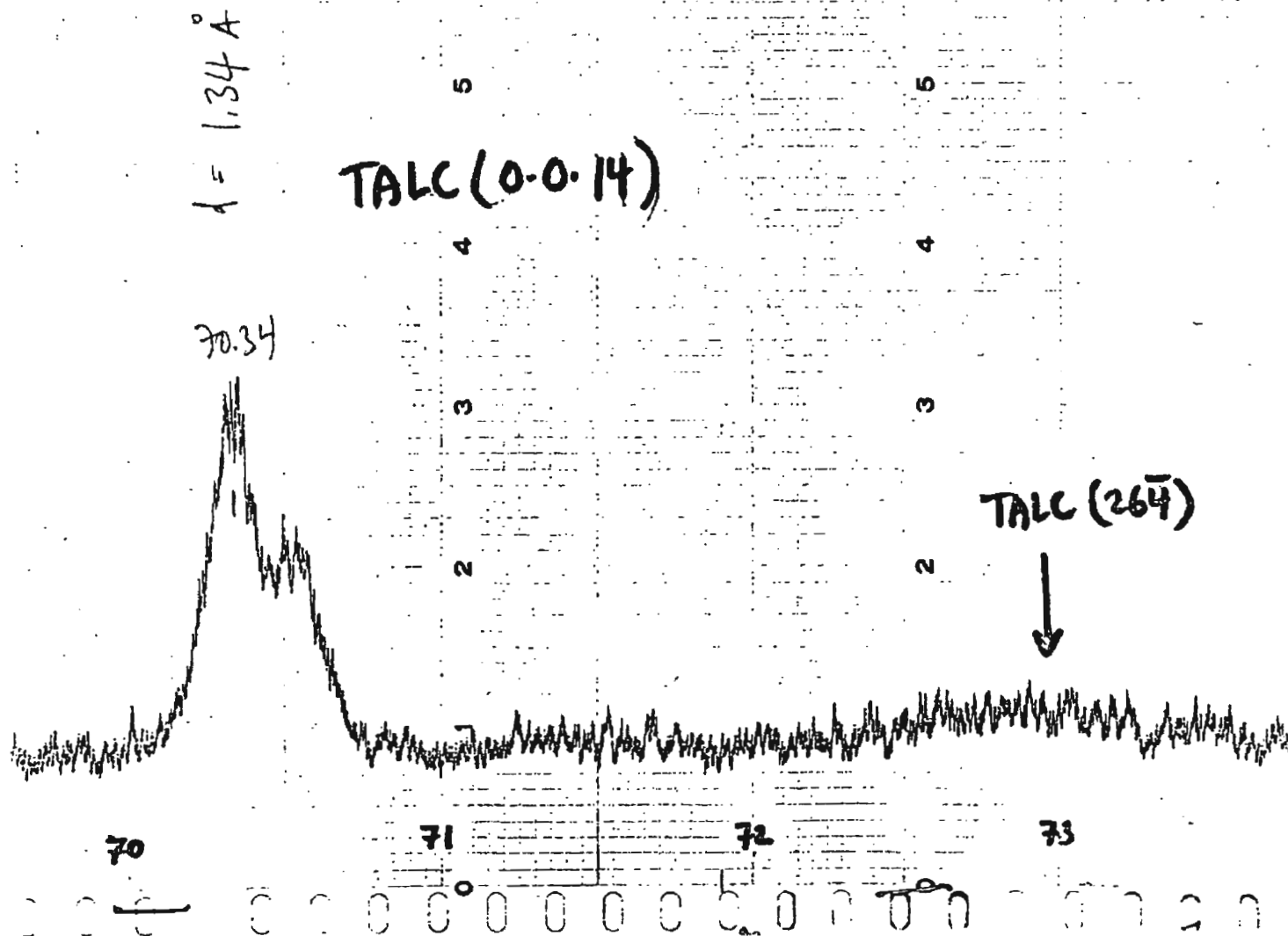


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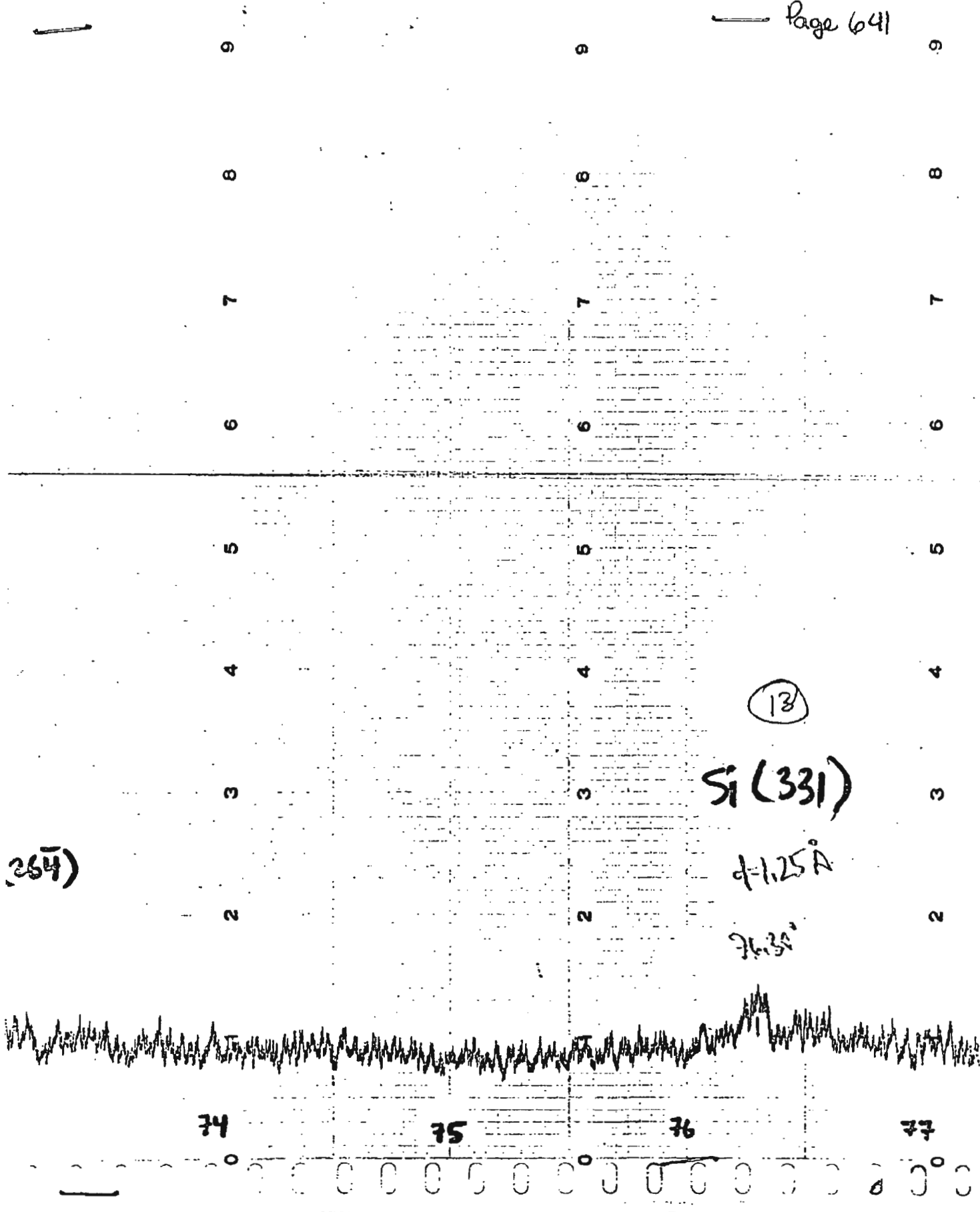
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1

2 3 4 5 6 7 8 9

2 3 4 5 6 7 8 9

LEWIN'S SHAWEL TO SHAWEL (BEFORE H₁)

1/4 20/min 40KV 20mA SCALE FACTOR=4
TIME CONSTANT=2

BASE=2U

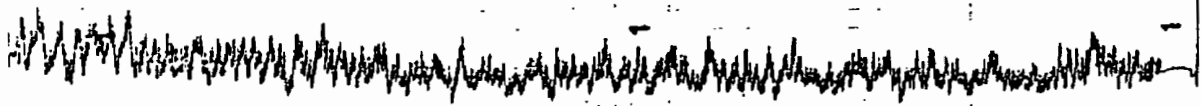
Δ = 2U

Lewins Shawel to Shawel
Before HCL Treatment

Page 642

80°-5'20" SCAN

< 80° 20' 1/4 20/gim.



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80

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Critique



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Comments on S.Z. Lewin's Presentation at the FDA- Johnson & Johnson
Meeting (9/21/72)

That Professor Lewin is a polished speaker is not questioned after his presentation on the morning of Sept. 21; however, his interpretation and command of the theory of x-ray diffraction and optics is suspect. After a somewhat theatrical and generally non-relevant discourse on the structures of talc and chlorite, he claimed that Shower-to-Shower did indeed contain chrysotile. It appears difficult for Professor Lewin to decide on a fixed amount of chrysotile (his estimates vary from 2-5% on a given date) or whether tremolite is present. The diffraction patterns of sample numbers 136 and 137 handed out by Lewin are totally useless in deciding almost anything except the presence of talc and chlorite. On the other hand they do show, if marked correctly, that a β -filter was not used, a dangerous practice in general which only complicates an x-ray pattern. They do not show evidence of chrysotile or tremolite. His interpretation of the photomicrographs demonstrated a seeming lack of experience and little knowledge of the optical properties of talc. It is not uncommon for talc edges to be rolled or bent during mechanical grinding. This would explain Lewin's observation that some of the talc plates display two different refractive indices which he interpreted as indicating talc intergrown with chrysotile. According to Deer, Howie and Zussman (1962) the refractive indices of talc are $n_x \approx 1.54$, $n_y \approx 1.590$, $n_z \approx 1.590$. When a talc plate has rolled edges, one of the measured indices for the rolled edge will be near 1.54 with the unrolled plate exhibiting indices near 1.590. Lewin also claimed that parallel extinction of these edges and other fibrous-looking particles precludes their being talc. Talc always shows parallel extinction when viewed parallel to the plates or when rolled into fibrous-looking tubes. His claim that the "intergrown chrysotile" represents a new variety with different physical properties and structure from the normal fibrous variety is totally without sound scientific evidence. No such discovery has been claimed in the rather extensive literature on talc and chrysotile. Neither Lewin's x-ray data nor his optical data supports his claim that chrysotile is present in Shower-to-Shower.

Gordon E. Brown


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W. C. McCrone

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646

21 August 1972

Dr. A. Goudie
Johnson & Johnson
Research Center
501 George Street
New Brunswick, New Jersey 08901

Dear Al:

I thought that before we met with Dr. Brown at Princeton you and he might wish to see the x-ray data that we had produced on your Shower-to-Shower products. I am therefore enclosing a preliminary report on this work, together with Xerox copies of all the x-ray traces.

I would also like to thank you for the very complimentary letter which you sent to Dr. McCrone about our work on this project. Although you aimed your compliments specifically at me, I take it that you really mean the team as a whole. Without the work that Gene Grieger and Ralph Hinch put in, particularly on the midnight shift on the night of the 10th, any contribution that I could have made to the FDA meeting would have been only superficial. Finally, a lot of the credit for the FDA presentation must go to the J & J people who, as ever, were helpful, courteous and encouraging while the work was in process.

Yours sincerely,

Ian M. Stewart
Senior Research Physicist

IMS:ajw
Ref: MA 2540
Enclosure

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PRELIMINARY NOTES ON THE EXAMINATION OF
SHOWER-TO-SHOWER PRODUCTS FOR JOHNSON AND JOHNSON

Because of allegations that a sample of Johnson & Johnson's Shower-to-Shower talcum powder contains approximately 4% chrysotile, several samples of this product have been examined by x-ray diffraction, electron microscopy and, in some cases, optical microscopy.

At the start of this investigation the sample alleged to contain chrysotile was not available and therefore 15 samples covering the production span during which this sample would have been manufactured were examined by x-ray diffraction and electron microscopy. Neither technique revealed any evidence of chrysotile. Later, the sample which had been alleged to contain chrysotile was obtained and this was similarly examined: again no chrysotile was detected. Two additional samples, identified as sample BEG and sample MID, representing the start of the production run and the middle of the production run from which the alleged chrysotile containing sample was manufactured, were also examined and neither of these showed chrysotile. These last three samples were also examined petrographically and again no chrysotile was found.

To comment briefly on our results in comparison with those of Professor Lewin in the talc samples which we looked at we do have a doublet which occurs very close to the chrysotile line and it is possible that Lewin confused one of these peaks with the chrysotile peak. However, if this is the case I would suspect that the level of chrysotile, if you really computed it on this peak, would show as very much higher than the figure he is quoting. Furthermore, as you know, we did not detect the higher order chrysotile line which we maintain should be present and which Lewin says is absent due to preferred orientation. Preferred orientation would not eliminate this line, since it is a fourth order reflection (004) from the same planes of which the 12. 210 20 is a second order reflection (002). Thus it is clear that the argument of a preferred orientation is falacious. To further substantiate that this line was not chrysotile, we ran both a chrysotile standard and our sample number 6 (the batch dated 3-2-71) which we spiked with 5% chrysotile and clearly show a third peak occurring between

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the previously mentioned doublet. Thus we are satisfied that Lewin's interpretation is erroneous. I would stress that our argument with Lewin is on interpretation: the x-ray diffraction curves which he has agree completely with those which we have.

Finally, we also ran powder diffraction samples using a 57.3 mm camera on our chrysotile standard and on the same talc number 6 and were unable to find chrysotile in sample number 6, although previous work with you had indicated that we were picking it up by photographic techniques at around the 1-2% level.

In conclusion, then, we have identical x-ray diffraction data to those of Professor Lewin which in our opinion show no evidence of chrysotile. To support our opinion the facts we present are firstly, the absence of a higher order reflection from the same plane as that on which Professor Lewin bases his identification; secondly, when a sample is intentionally spiked with chrysotile the presence of a new line near the peak on which Professor Lewin bases his conclusion together with its higher order reflection; thirdly, the absence of any evidence of chrysotile by either light microscopy or electron microscope combined with electron diffraction.

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JNJAZ55_000017759

COMMENTS ON DR. LEWIN'S TECHNIQUE

I will consider only his optical crystallographic techniques. As I understand Dr. Lewin he treats talc and asbestos as though they are both isotropic. Although this is not so in either case, he justifies this action on the basis that talc in the plate view and asbestos in any orientation has low birefringence — talc: 1.594–1.600 or 0.006; chrysotile: 1.549 – 1.556 or 0.007). This is not too bad but he uses a refractive index liquid, 1.580, to differentiate between these two compounds. If an apparent needle shows an index less than 1.580 it is chrysotile.

Yet, if talc plates are tipped only a few degrees, say 20–30°, the index of talc is no longer 1.594–1.600 but in one orientation relative to the plane of vibration of polarized light is less than 1.580.

In other words, talc plates on edge are "chrysotile" by Dr. Lewin's procedure. If, however, he simply rotated the stage 90° chrysotile would miraculously revert to talc. Every crystal of talc will always show one refractive index (in one orientation relative to the polar) higher than 1.580. It will show both indices higher than 1.580 only when lying flat or nearly flat. Chrysotile will never show an index above 1.580.

Dr. Lewin also uses birefringence to differentiate between chrysotile and talc. He seems to feel that talc always shows low birefringence and asbestos high birefringence. This is not so. The birefringence of talc is low when viewed perpendicular to the plate but high (much higher than chrysotile) when the plate is tipped. The figures are:

	N_2	N_1	$N_1 - N_2$
Talc (plate view)	1.600	1.594	0.006
Talc (on edge)	1.600	1.550	0.050
Chrysotile	1.556	1.449	0.007

Conclusion—"a little bit of knowledge is a dangerous thing."

Walter C. McCrone

Walter C. McCrone
Scientific Advisor

J&J-0130740



walter c. mccrone associates, inc.

CONSULTING: ULTRAMICROANALYSIS • MICROSCOPY • SMALL PARTICLE PROBLEMS • SOLID-STATE CHEMISTRY

650

29 September 1972

Dr. A. J. Goudie
Johnson & Johnson
Research Center
501 George Street
New Brunswick, New Jersey 08901

Dear Dr. Goudie:

Here are the comments you asked for on Professor Lewin's presentation at the last FDA meeting held on 21 September 1972. My remarks must naturally be made in the context of what occurred at previous meetings. I have also attached Dr. McCrone's comments.

X-ray Diffraction

At the early FDA meeting Professor Lewin had stated that chrysotile was present in your Shower to Shower product at about the 5% level. You will recall that there was some dispute between him and myself regarding the interpretation of this data. Basically the difference of opinion is that Professor Lewin was identifying chrysotile on the basis of one line which occurs at a 2θ value of approximately 12° and I maintain that when this line is present it should be accompanied by a line of approximately equal intensity at the position of approximately $24.6^\circ 2\theta$, the latter line being a second order reflection of the first line and both of them being due to basal planes in the system. In the nomenclature of x-ray crystallography, the first line is referred to as a (002) line and the second a (004). Dr. Lewin, however, had claimed the absence of the (004) line in his data on the basis of preferred orientation, an argument that is clearly preposterous: it would be equivalent to saying that opposite faces of a cube can be non-parallel and the cube would still be a cube.

At the second meeting Professor Lewin seemed to deemphasize his x-ray data. The amount of chrysotile which he was quoting for your material had been revised to approximately 2%. He still had not step-scanned the peaks concerned. He still does not have the higher order reflection showing. I think the point that Professor Bueger raised—that at least 3 lines are necessary for the identification by x-ray diffraction of a phase present—is important, but I think equally important in this case is the fact that the absence of the (004) reflection means that we certainly do not have chrysotile. During the whole meeting, however, Professor Lewin played his x-ray diffraction data in very low key and in fact I cannot recall him mentioning it, except in reply to questions from other people.

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Dr. A. J. Goudie
Page Two

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Light Microscopy

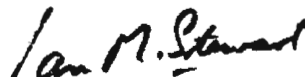
I think Professor Lewin's light microscopy is a little suspect. I do not think he has been sufficiently critical of his observations and I do not think that he has fully appreciated the optics of the serpentine and talc minerals. He repeatedly referred to the refractive index of talc where he clearly meant the mean refractive index of a plate section, but completely ignored the refractive index normal to the plates. From our electron microscopy, our own light microscopy and our scanning electron microscopy, we know that there are thin shards and ribbons of talc which can be seen on edge in many sections. These I think are what Professor Lewin is calling chrysotile. What adds credence to my belief is that he refers to the chrysotile as being quite obvious on the basis of its high birefringence. However, the birefringence of chrysotile is less than 0.01 whereas the birefringence of a talc plate, if observed on edge, is 0.05, thus his high birefringence particles are more likely to be talc. Many of the particles which he calls chrysotile occur on talc plates and it is noticeable that many of those are parallel to the edges of the plate. This is fairly typical of cleavage steps on a cleavage plate which are curling at the edges and I expect he has been misled by this.

Chrysotile Formation

Professor Lewin's speculation on the formation of chrysotile within the talc plates is extremely interesting. In one fell swoop he has completely reversed the normal order of petrogenesis. Talc may form in three ways; one of these ways is not clearly understood and is the development of talc in rocks which are high in gypsum. The second and third ways involve the formation of talc from other rocks by hydrothermal solutions. These are the two main types of talc formation. The first one is by the further alteration of serpentine, the second is by the alteration of rocks containing a high proportion of calcite, dolomite and quartz. From the description given by Dr. Pooley, the latter mechanism is the operative one in this particular mine.

To postulate this "new mechanism" and "new type" of chrysotile with its x-ray and optical data also differing from the well characterized forms of chrysotile, I think is rather fatuous and an attempt to extricate himself from a position into which he should never have allowed himself to slip. My personal feeling is that when asked to do petrographic work on these samples he should have realized that he was getting further from his own field and should have recommended to the FDA that they place this aspect of the work with a competent mineralogist/petrologist. In this way all the parties involved could have been saved a lot of trouble.

Yours sincerely,



Ian M. Stewart
Manager, Electron Optics
Group

IMS:ajw
Ref: MA 2546
Enclosure

J&J-0130742

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Colorado School
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J&J-0130743

JNJAZ55_000017763

MR. T. CANEER

Born: USA

Education: B.A. Geology, University of Missouri

M.A. Geology, University of Missouri

Employment: Assistant Manager Mining Division and
Chief Mineralogist -Present
Colorado School of Mines Research Institute

COLORADO SCHOOL OF MINES RESEARCH INSTITUTE

P.O. Box 112

GOLDEN, COLORADO 80401

653

September 7, 1972

Dr. Bob Rolle
Johnson & Johnson
West Research
501 George Street
New Brunswick NJ 08903

Dear Dr. Rolle:

In compliance with a request from Dr. Goudie I have made X-Ray diffraction studies on the sample of talc product (shower to shower) which he sent. The objective of these studies was to determine the nature of a reported double X-Ray diffraction peaks in the vicinity of 7 to 7.5 angstroms. More specifically he wanted to determine if the reported data resulted from the occurrence of chrysotile. The enclosed X-Ray diffraction scan for the sample indicates the presence of talc and chlorite. Since the major peaks of chrysotile occur near the chlorite peaks, step scanning was used in order to better determine if any chrysotile peaks occur. The enclosed three charts show the results of these scans. I first scanned the vicinity of the second order chlorite peak (7.0-7.2 angstroms) since a major chrysotile peak occurs at about 7.4 angstroms. As may be seen on chart 1, a chlorite peak occurs at 7.1 angstroms. This peak contains a shoulder which occurs at about 7.22 angstroms. The position of this peak indicates that it is also a chlorite peak (the variety of chlorite called penninite contains a diffraction peak in this vicinity). Chrysotile should give a peak representing about 7.36 angstroms (about $12.0^{\circ}2\theta$). However, realizing that X-Ray diffraction equipment alignment and compositional variations are critical, the area around the first order chlorite peak (6.0 to $6.5^{\circ}2\theta$) was also step scanned. If the 7.22 angstrom peak (shown in chart 1) results from a second order chlorite peak then its first order peak should occur at about 14.4 angstroms. Chrysotile does not have a peak of these dimensions. As may be seen in chart 2, a major peak representing 14.1 angstroms occurs which is the first order peak of the major chlorite peak shown in chart 1 (at 7.1 angstroms). However, this peak also has a shoulder which occurs at about 14.47 angstroms. This indicates that the shoulder ($7.22\text{\AA}$ or $12.25^{\circ}2\theta$) shown in chart 1 is probably chlorite and not chrysotile.

In order to be more complete, the region in the vicinity of 3.7 to 3.5 angstroms (24° to $25.5^{\circ}2\theta$) was step scanned. As shown on chart 3 two peaks occur. One at 3.54 angstroms, which is the fourth order of the 14.1 angstrom chlorite peak. The other occurs at $3.61\text{\AA}$ which would be the fourth

Mineral Industry Research

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Dr. Bob Rolle

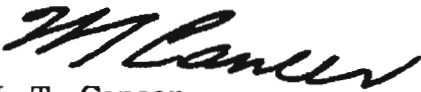
Page 2

September 7, 1972

order of the $14.47 \text{ \AA}$ peak which is also likely to be chlorite. No peak was observed in the vicinity of 3.66 angstroms where the ASTM file card shows the fourth order chrysotile peak.

The above data does not indicate the presence of chrysotile.

Sincerely,



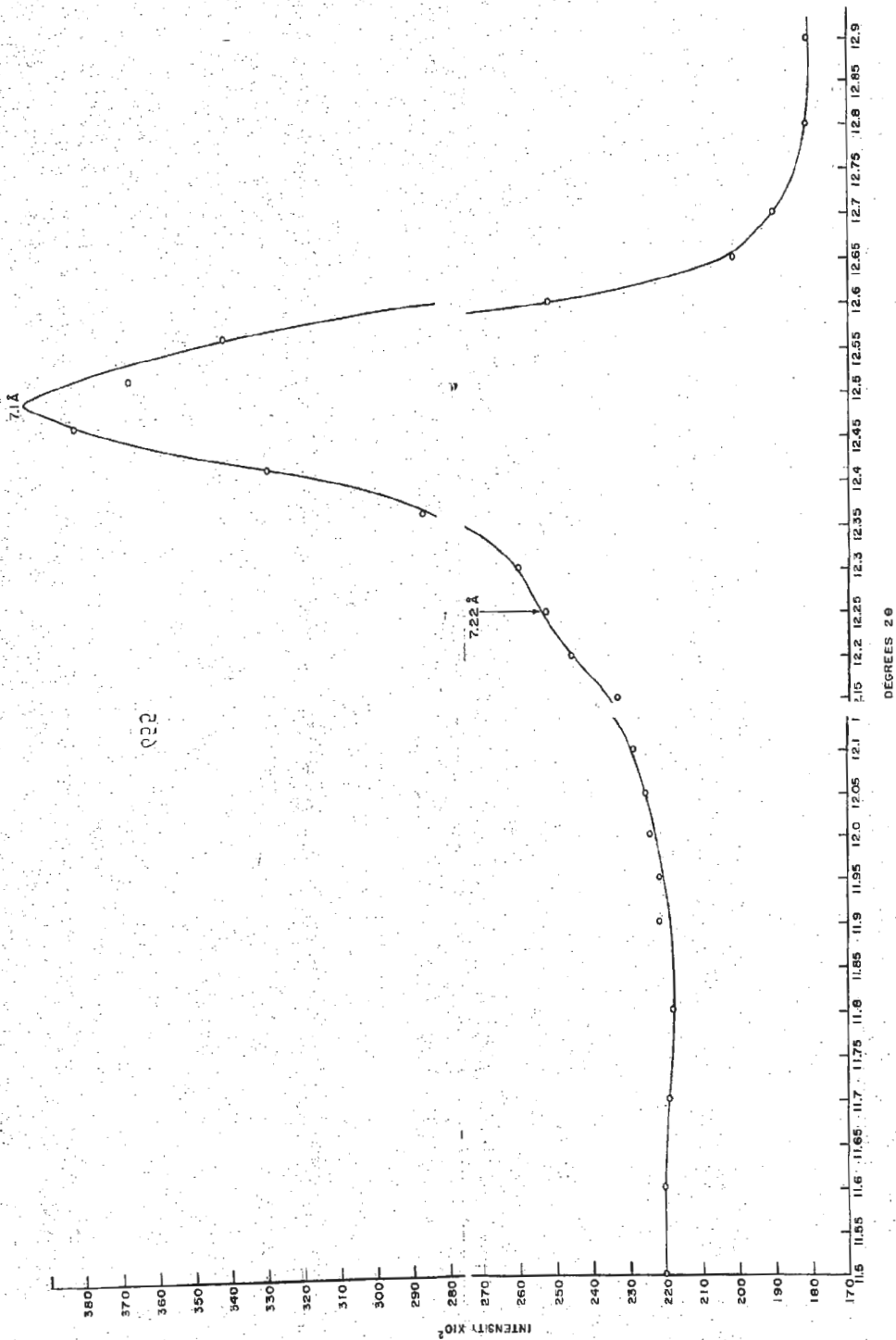
W. T. Caneer
Assistant Manager
Mining Division

WTC/psk

cc: Mr. Bill Ashton (without enclosures)
Dr. Goudie (with enclosures)

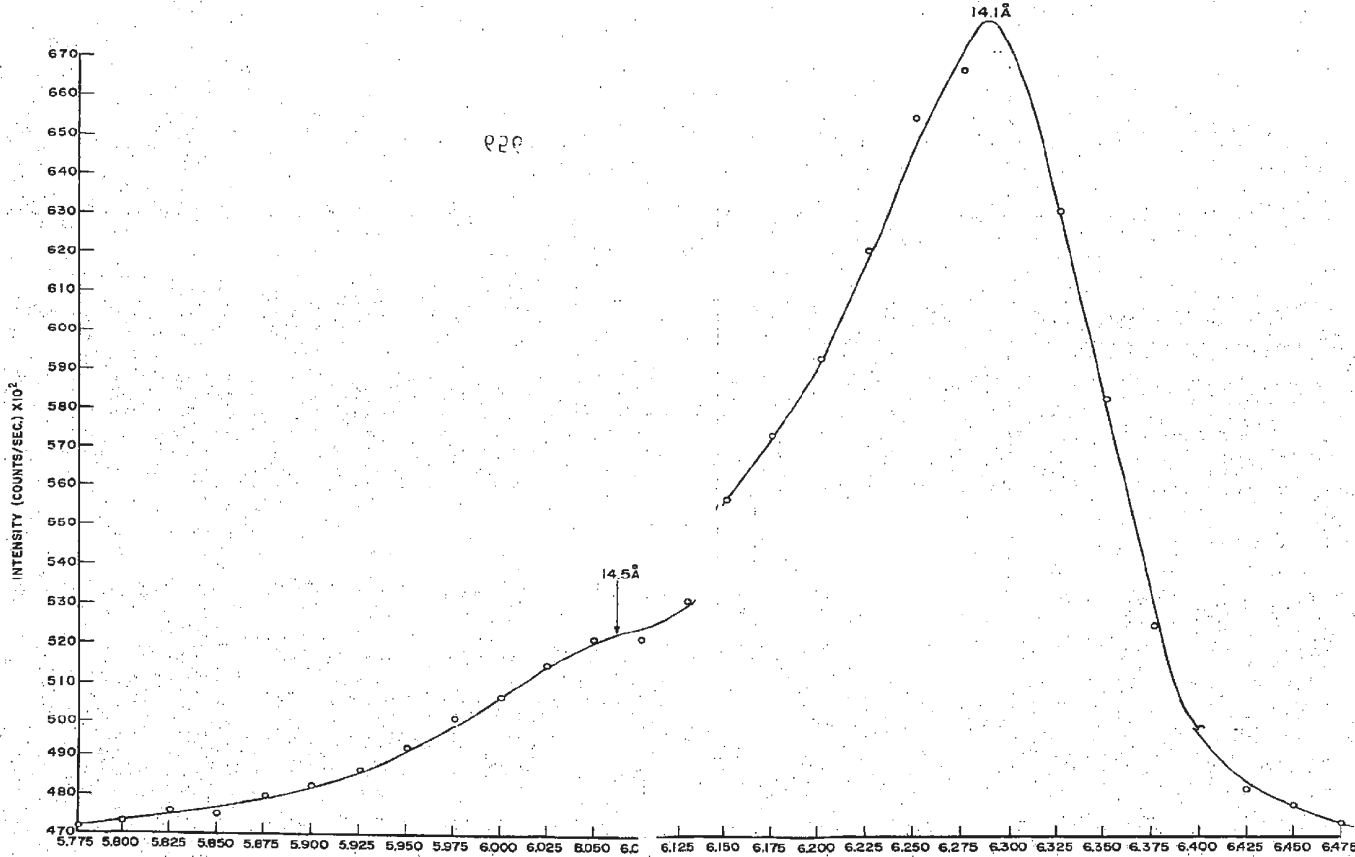
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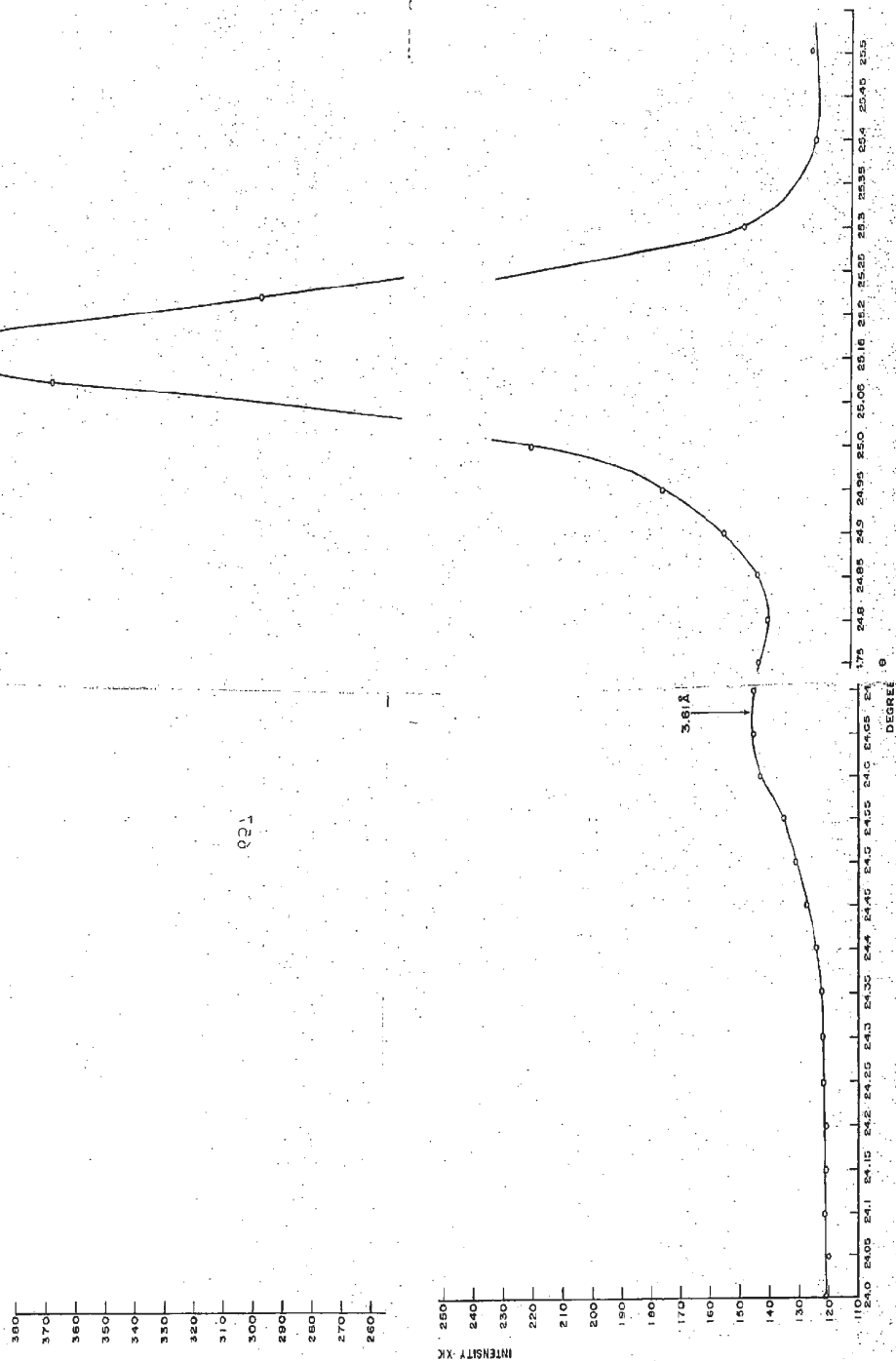


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Critique

J&J-0130750

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COLORADO SCHOOL OF MINES RESEARCH INSTITUTE
P.O. Box 112
GOLDEN, COLORADO 80401

658

October 2, 1972

REFER TO

C10704

Dr. A. J. Goudie
Director, Analytical and
Sterilization Section
Johnson & Johnson
West Research
501 George Street
New Brunswick NJ 08903

Dear Dr. Goudie:

I will first comment on my impressions of Dr. Lewin's presentation before the FDA board. I got the impression that Dr. Lewin was trying to be expert in perhaps too many fields. As a result he had only enough knowledge in many of the scientific fields to confuse the issue -- the identification of chrysotile in talc. I must add, however, that he does give quite a convincing presentation if one has only a little, or, no knowledge of mineralogy.

One example of his lack of knowledge in mineralogy was related to x-ray diffraction identification of minerals. He wasn't aware of the fact that chlorite occurs in more than one variety. He apparently has been indicating that chrysotile is present in a sample by wrongly using chlorite x-ray diffraction data.

He was apparently very confused relative to optical microscopy based on his ideas on indices of refraction. He also stated to me that atomic packing or density had no bearing on indices of refraction. This is not so.

I will next comment on the FDA meeting in general. Since the accepted FDA method for determining if chrysotile is present in talc is by first screening with x-ray diffraction. If the presence of serpentine is suspected then the sample is subjected to optical examination. The FDA is aware that x-ray diffraction will not pick up traces of chrysotile (serpentine). They are probably also aware that if one looks long enough by optical methods he may find a fiber of chrysotile in any talc (or in anything else) which may have resulted from contamination from the air. Such a stray fiber should not condemn talc. However,

Mineral Industry Research

J&J-0130751

Dr. A. J. Goudie

Page 2

October 2, 1972

under the present scheme of things it apparently would be enough to cause a ban of the talc. The screening of talc by x-ray diffraction provides a method for not allowing this to happen, since this method is not that sensitive. My point in this is that Dr. Lewin apparently finds cause to go to optical methods based on the observation of a 7Å peak from x-ray diffraction. As I pointed out at the last FDA meeting, the 7Å peaks in the Italian talc were caused by chlorite. Since practically all talc contains chlorite, the x-ray diffraction screening will be negated if one just looks for a 7Å peak by x-ray scanning. I suggest that chlorite is checked for by x-ray diffraction when a 7Å peak is encountered before going to optical methods. This should be done by step scanning the 7Å and 14Å regions. If a 14Å peak is found corresponding to the 7Å peak then they both belong to chlorite. Thus there is no need to go to optical methods.

Sincerely,



W. T. Caneer
Assistant Manager
Mining Division

/laj

cc: Wm. H. Ashton

J&J-0130752

Prof. F. D. Pooley

J&J-0130753

JNJAZ55_000017773



University College, Cardiff

Postal Address: University College, Newport Road, Cardiff CF2 1TA.
Telephone Cardiff 442111 Telegrams: Coleg Cardiff

From: Dr. F.D. Pooley
Department of Mineral Exploitation

660

Examination of 'Shower to Shower' Talc Sample for Johnson and Johnson

The 'Shower to Shower' talc specimen was subjected to electron microscope examination, X-ray analysis and also to density separation tests.

The density separation tests were performed in a heavy organic liquid composed of a mixture of tetrabromoethane and carbon tetrachloride. The density of the fluid was set at an S.G. of 2.5. being intermediate between the density of chrysotile and talc. Visually all the sample suspended in the liquid was found to sink indicating the sample had a higher density than 2.5. An examination of the visually clear solution at the top of the separating vessel showed that no particles were present, thus indicating no free particulate material with a density below 2.5 thus precluding the presence of chrysotile.

X-ray examination of the specimen indicated that no amphibole asbestos at the level of detection of the X-ray technique (i.e. 1.0%) was present. Electron microscope examination of the sample revealed no chrysotile fibres in the sample. Samples of 'Shower to Shower' talc diluted with chrysotile at the 1 and 5% levels showed that if chrysotile were present it should be detected at below the 0.05% level by Electron microscope examination.

The conclusions to be drawn from the examination are that no detectable chrysotile or amphibole asbestos is present in the 'Shower to Shower' talc sample.

F.D. POOLEY

27th September, 1972.

J&J-0130754

JNJAZ55_000017774

Critique

J&J-0130755

JNJAZ55_000017775



University College, Cardiff 661

Postal Address: University College, Newport Road, Cardiff CF2 1TA.
Telephone Cardiff 442111 Telegrams: Coleg Cardiff

From..... Dr. F.D. Pooley
Department of..... Mineral Exploitation

FDP/MM

29th September, 1972.

Dr. A.J. Goudie,
Director,
Analytical Research Section,
Johnson and Johnson,
New Brunswick,
New Jersey,
U.S.A.

Dear Dr. Goudie,

The results presented by Dr. Lewin at the F.D.A. meeting would appear not to be capable of substantiating the presence of quantities of chrysotile asbestos. The X-ray procedure adopted was shown to be very insensitive to the detection of 1% chrysotile, while the major identification of chrysotile was based only on the observation of a slight difference in refractive index, which could have been explained by variation in orientation of talc particles or the presence of another mineral. As the refractive index of the unknown material appeared to be lower than that of talc, it was stated that chrysotile was present because it had a slightly lower refractive index. However, so have many other minerals, including magnesite which is a well known contaminant of talc.

RI talc	1.539	1.589	1.589
	-1.550	1.594	1.600
RI chrysotile	1.532	-	1.545
	1.549		1.556
RI magnesite	1.417	-	1.507
	1.523		1.545:

Variation in refractive indices therefore only indicates an orientation difference or perhaps the presence of another mineral phase.

J&J-0130756

JNJAZ55_000017776



University College, Cardiff

662

Postal Address: University College, Newport Road, Cardiff CF2 1TA.
Telephone Cardiff 442111 Telegrams: Coleg Cardiff

From Dr. F.D. Pooley
Department of Mineral Exploitation

The positive identification of chrysotile can only be made with a transmission electron microscope because it is with this instrument that the fibre can be observed and defined. The name Chrysotile and Chrysotile asbestos belong only to that material with a fibrous structure composed of fibrils 250-350 Angstroms in diameter if these are not observed in a sample it must be concluded that the material is free of Chrysotile.

It is suggested, therefore, that talc samples should be examined by a mineralogical laboratory with transmission electron microscope facilities as it has already been shown to be the only way of detecting and assessing chrysotile in other materials. It would also be interesting to see whether the amounts of supposed chrysotile detected by Dr. Lewin do in fact vary with carbonate mineral contamination of the talc samples.

DR. F.D. POOLEY

J&J-0130757

JNJAZ55_000017777

Prof. S. Pollack

J&J-0130758

JNJAZ55_000017778

POLLACK, DR. SIDNEY S(OLOMON), b. New York, N.Y. Sept. 24, 29;
m. 53; c. 2. CRYSTALLOGRAPHY. B.S, Mich. State, 51, M.S. 53;
Ph.D. (soil sci), Wisconsin, 56. FEL. X-RAY DIFFRACTION, MELLON
INST, 56- Soil Sci. Soc; Mineral. Soc; Crystallog. Asn. Clay
mineralogy; polymers; low crystallinity carbons; small-angle x-ray
diffraction and crystallography; meteorites. Address: Mellon Institute,
4400 Fifth Ave, Pittsburgh, Pa. 15213.

From: American Men of Science, 1966

J&J-0130759

JNJAZ55_000017779

Carnegie-Mellon University

Mellon Institute
4400 Fifth Avenue
Pittsburgh, Pennsylvania 15213
[412] 621-1100

Division of Educational and Research Services

September 27, 1972

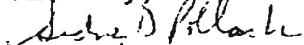
Dr. A. Goudie
Johnson and Johnson
Research Center
501 George Street
New Brunswick, New Jersey 08901

Dear Dr. Goudie:

The meeting with the representatives of FDA was an enlightening experience for me and I am happy to give you some of my impressions from the meeting. Although Dr. Lewin said that chrysotile was present in two samples, he presented no objective evidence. Since x-ray diffraction cannot be used to make a positive identification of asbestos when it makes up less than about three percent of a sample, the combination of electron micrographs showing a hollow fiber, and then the characteristic electron diffraction pattern from the fiber, make the most definitive identification. Dr. Lewin did not show these. Optical microscopy is another sensitive technique for identifying very small amounts of asbestos but no evidence of crystals with the indices of refraction characteristic of chrysotile was presented. Dr. Lewin's identification of asbestos appeared to be based on the presence of particles which he thought looked like asbestos. He could not support his identification with any of the necessary electron microscopy, electron diffraction or optical microscopy evidence.

To me, Dr. Lewin indicated his lack of fundamental understanding of crystallography at the very beginning of his presentation when he showed a scale model purporting to be the mineral talc. In this model, he had the basic platy units of talc which are about 10 Angstroms thick, 20 Angstroms apart because the c unit cell is about 20 Angstroms. Dr. Lewin didn't understand that the manner in which the platy units pack and not the distance between them determined the c unit cell dimension. It is disconcerting that the many capable government mineralogists were not consulted about this problem.

Sincerely,



S. S. Pollack, Fellow

X-Ray Diffraction Laboratory

SSP:jdf

J&J-0130760

Carnegie-Mellon University

Mellon Institute
4400 Fifth Avenue
Pittsburgh, Pennsylvania 15213
[412] 621-1100

Division of Educational and Research Services

September 28, 1972

Dr. A. Goudie
Johnson and Johnson
Research Center
501 George Street
New Brunswick, New Jersey 08901

Dear Dr. Goudie:

The meetings with the people from Johnson and Johnson, the consultants and FDA were very educational and interesting. It is really hard to believe that Lewin still clings to the notion that there is asbestos in the talc.

I did not carry out any significant experimental work, but based on all the x-ray evidence, I would agree that there is no positive evidence for the presence of chrysotile in the SHOWER TO SHOWER product. Since the least detectable amount is 1-3%, this means that the x-ray diffraction says there is less than 1-3% serpentine. The nice DTA work indicates that there really is less than 1% chrysotile. In my view, the transmission electron microscopy and electron diffraction evidence are the best indicators that there is no chrysotile in the sample. The optical microscopy is also good evidence, but I suspect that in certain samples, one might have chrysotile fibers that could be seen using TEM and electron diffraction but not seen by optical microscopy.

I am glad that you felt my discussion the day before the FDA meeting was helpful and hope that I might be useful at some other time. It was a pleasure to meet the people from Johnson and Johnson and the other consultants.

Sincerely,



S. S. Pollack, Fellow
X-Ray Diffraction Laboratory

SSPjdf

cc: J. J. McGovern

J&J-0130761

J. P. Schelz
Johnson & Johnson

J&J-0130762

New Brunswick, N.J.
September 28, 1972

Subject: ANALYSIS OF SHOWER TO SHOWER* POWDER
FOR CHRYSOTILE ASBESTOS - PROJECT NO. 503

X-Ray Diffraction

Qualitative analysis by means of a continuous X-ray diffractometer scan was performed on the sample of SHOWER TO SHOWER Powder which had been analyzed by Dr. Lewin. The following mineral content was indicated:

Major: Talc
Minor: Chlorite
Trace: Dolomite, Calcite,
Magnesite, Quartz

In addition, an extraneous X-ray diffraction peak occurred at approximately $7.28\text{\AA}$, at very weak intensity. This peak was thought to indicate a trace amount of either a second variety of chlorite, or kaolinite. The X-ray diffraction step-scanning technique was then employed for the purpose of further identifying this trace mineral impurity. Figure 1 shows a duplicate determination of the exact position of the extraneous peak ($7.25\text{\AA}$). Step-scanning revealed an additional diffraction peak at $3.62\text{\AA}$ (Figure 2) and a shoulder at approximately $14.49\text{\AA}$ (Figure 3). The existence of these three peaks was accepted as confirmation that the unknown mineral impurity was, in fact, a second variety of chlorite.

It was desired to determine the minimum detectable level of chrysotile asbestos in talc by the X-ray diffraction technique. Homogeneous standards consisting of 1%, 3%, and 5% by weight of chrysotile in SHOWER TO SHOWER Powder were prepared and step-scanned in the vicinity of the chrysotile peak. The 5% sample (figure 4) showed a diffraction peak at $7.31\text{\AA}$ corresponding to chrysotile. This peak was easily detected in the 3% standard (Figure 5), but could not be detected at the 1% level.

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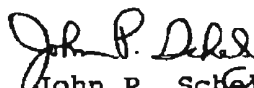
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The continuous X-ray diffractometer scans of SHOWER TO SHOWER Powder exhibited by Dr. Lewin indicate the absence of chrysotile in this talc. This is evident from the lack of diffraction peaks in the regions where chrysotile shows intense reflections ($7.31\text{\AA}$, $3.65\text{\AA}$).

Differential Thermal Analysis

A differential thermal analysis (DTA) method was developed for the detection and quantitative estimation of chrysotile asbestos in talc at low concentrations. This is possible because of the existence of the two intense thermal transitions of chrysotile which are free from talc interference. These transitions consist of a dehydroxylation endotherm with peak at 650°C . and an exothermic thermal decomposition peak at 820°C . (Figure 6). This thermal behavior is typical of chrysotile from sources all over the world. Thermograms of Italian talc (Figure 7) and SHOWER TO SHOWER Powder (Figure 8) indicate the presence of less than 5% each of chlorite and carbonate (mainly dolomite) impurities. Figures 9-11 show thermograms of standard samples of 5%, 3%, and 1% by weight of chrysotile in SHOWER TO SHOWER Powder. The arrows indicate chrysotile transitions. The 1% sample represents the minimum detectable level of chrysotile in talc by DTA. The thermogram of the SHOWER TO SHOWER Powder examined by Dr. Lewin (Figure 12) shows no thermal peaks in the temperature ranges characteristic of chrysotile; while 1% chrysotile added to this sample is readily detectable (Figure 13). On the basis of this experimental evidence, there is no chrysotile asbestos in SHOWER TO SHOWER Powder at a 1% level or greater.

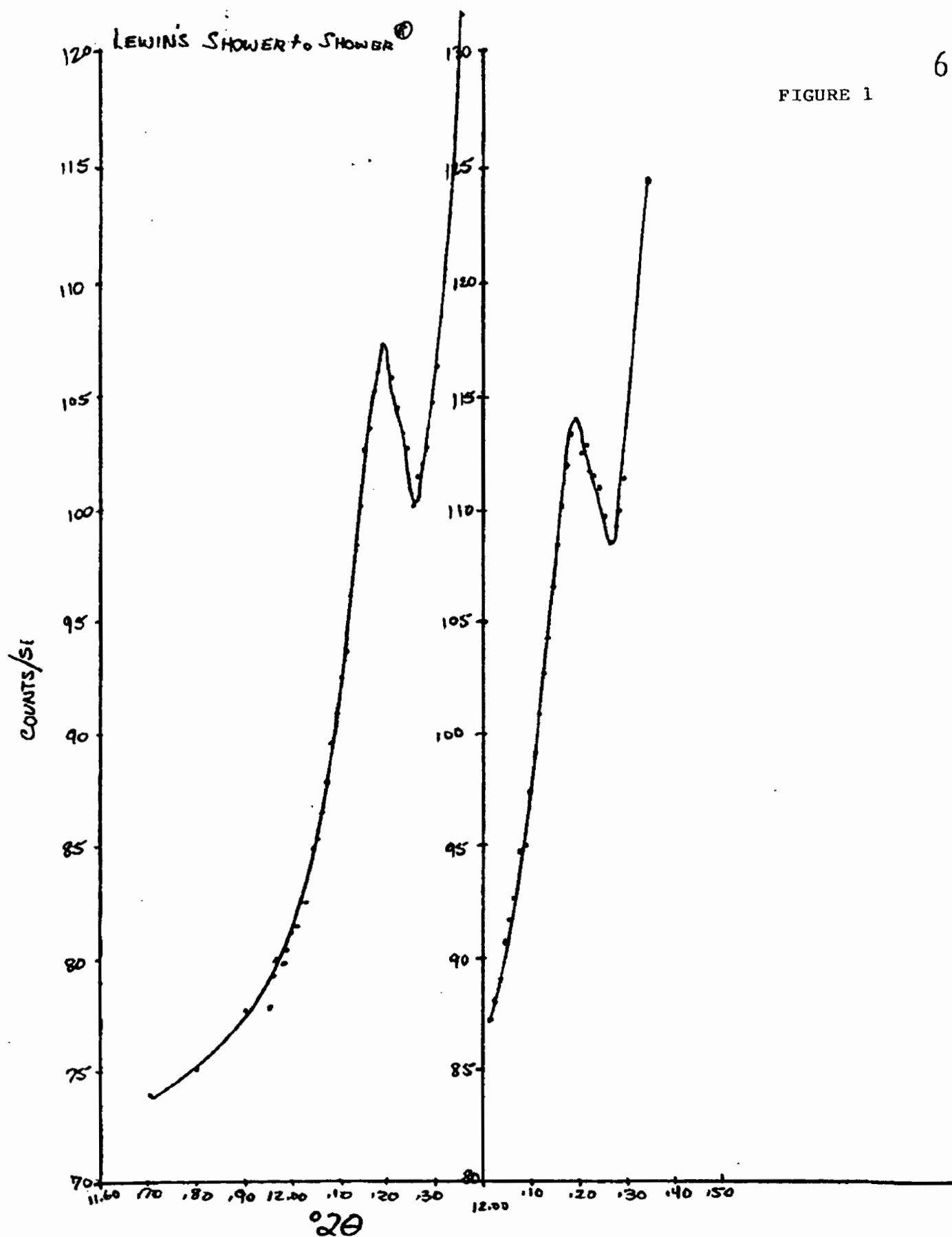
The theory of chrysotile fibers located within talc particles proposed by Dr. Lewin is erroneous with respect to detection of these fibers by DTA. The majority of the "fibers" detected by Dr. Lewin are located on the edges of talc platelets, not completely embedded in the talc particles. As such, and if these "fibers" were actually chrysotile, then they would of necessity undergo thermal transitions characteristic of the chrysotile structure. Since by DTA there is no chrysotile detected in the SHOWER TO SHOWER Powder at the proposed 2% level, the Lewin theory is shown to be in error.


John P. Scholz
Senior Research Scientist
Analytical Research

ab

J&J-0130764

FIGURE 1



J&J-0130765

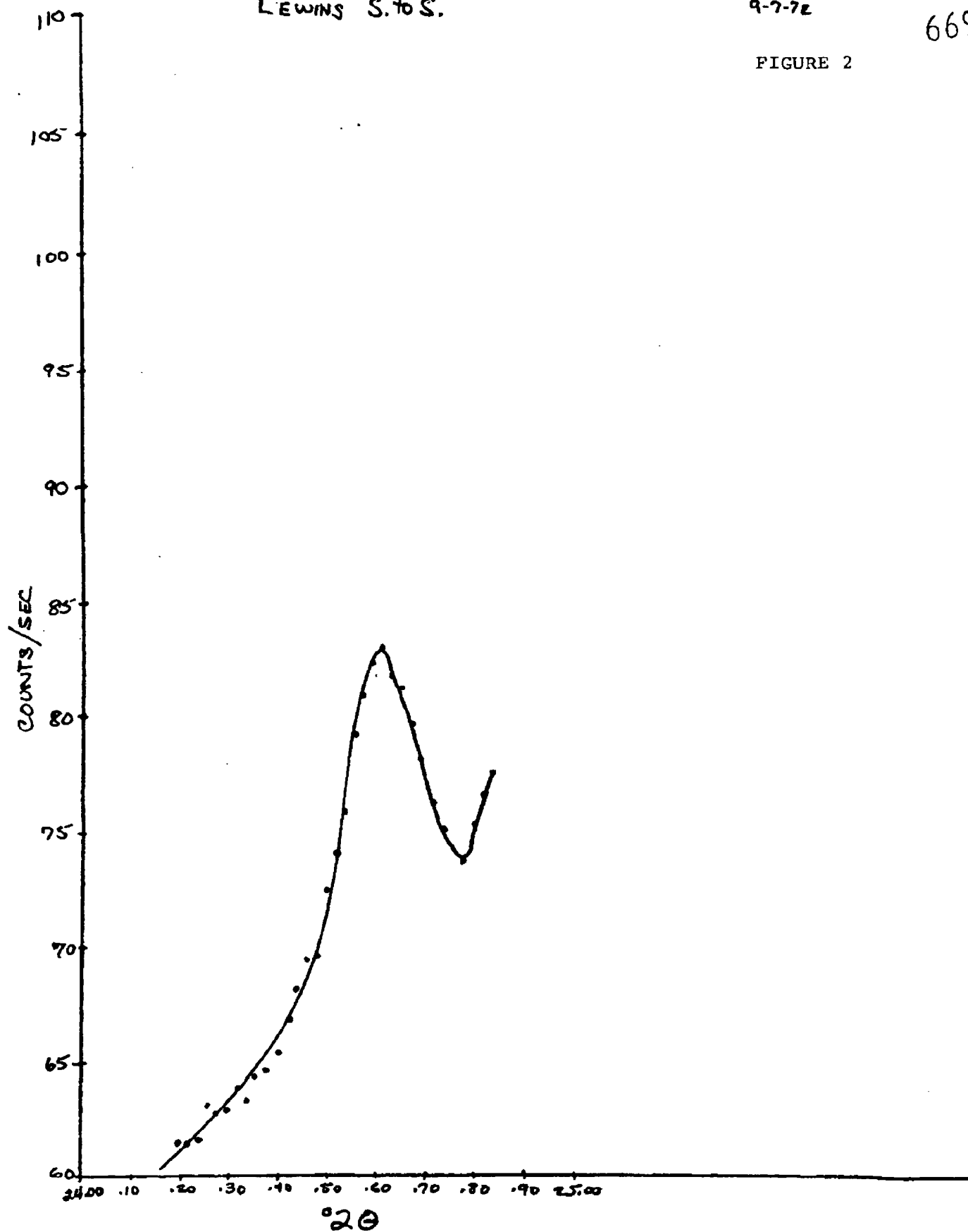
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LEWINS S.to S.

9-7-72

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FIGURE 2



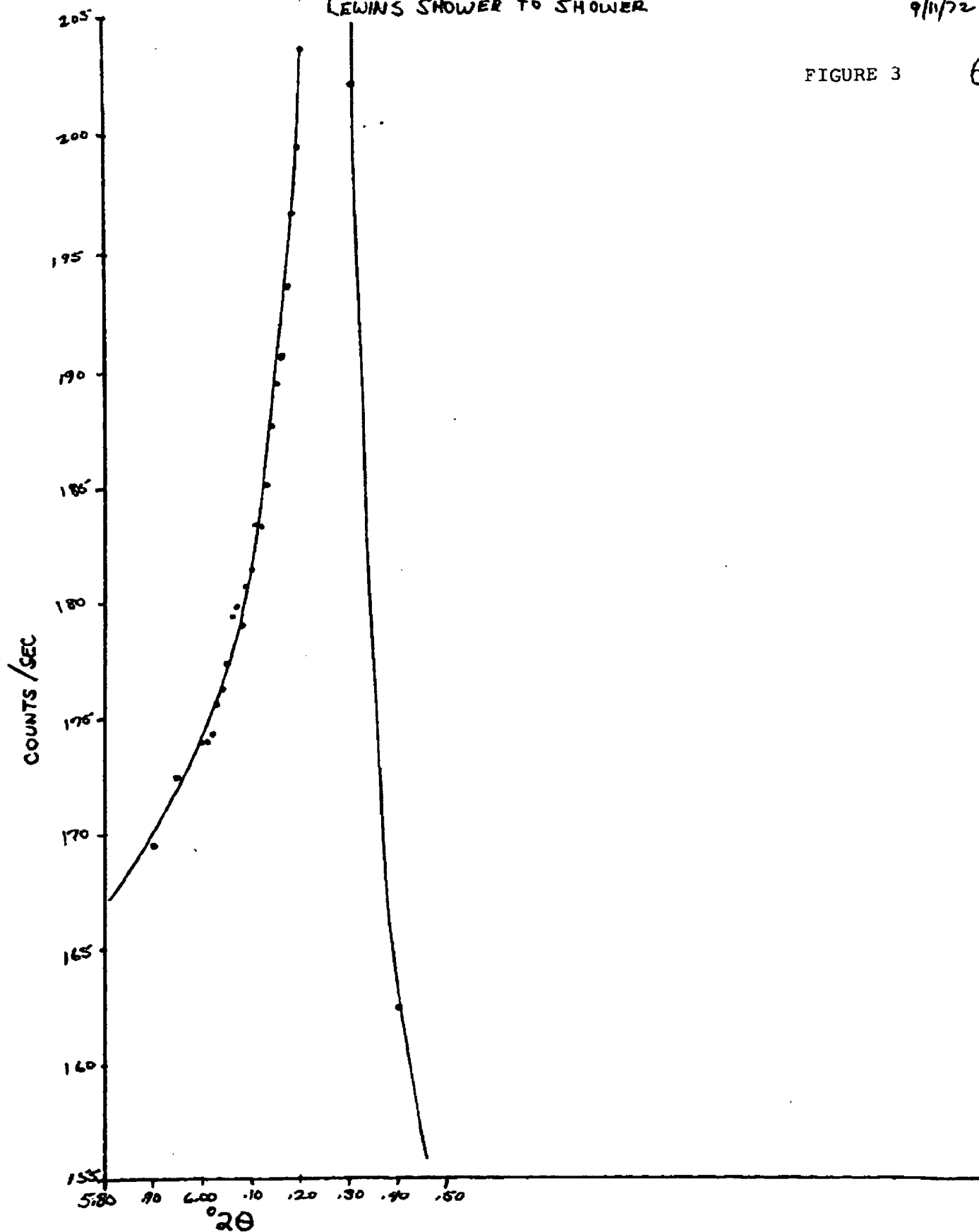
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LEWIN'S SHOWER TO SHOWER

9/11/72

FIGURE 3

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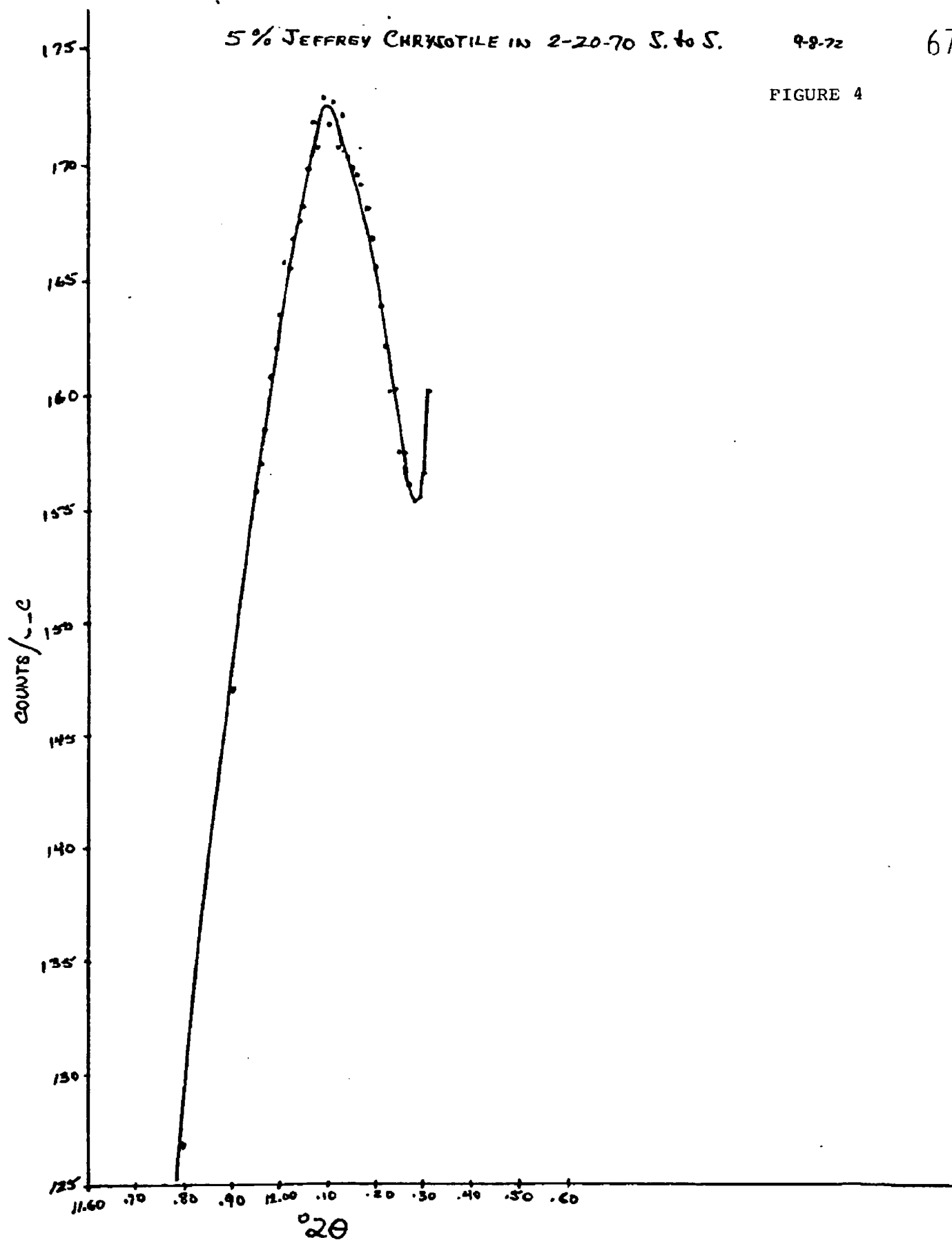
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5% JEFFREY CHRYSOTILE IN 2-20-70 S. to S.

9-8-72

671

FIGURE 4

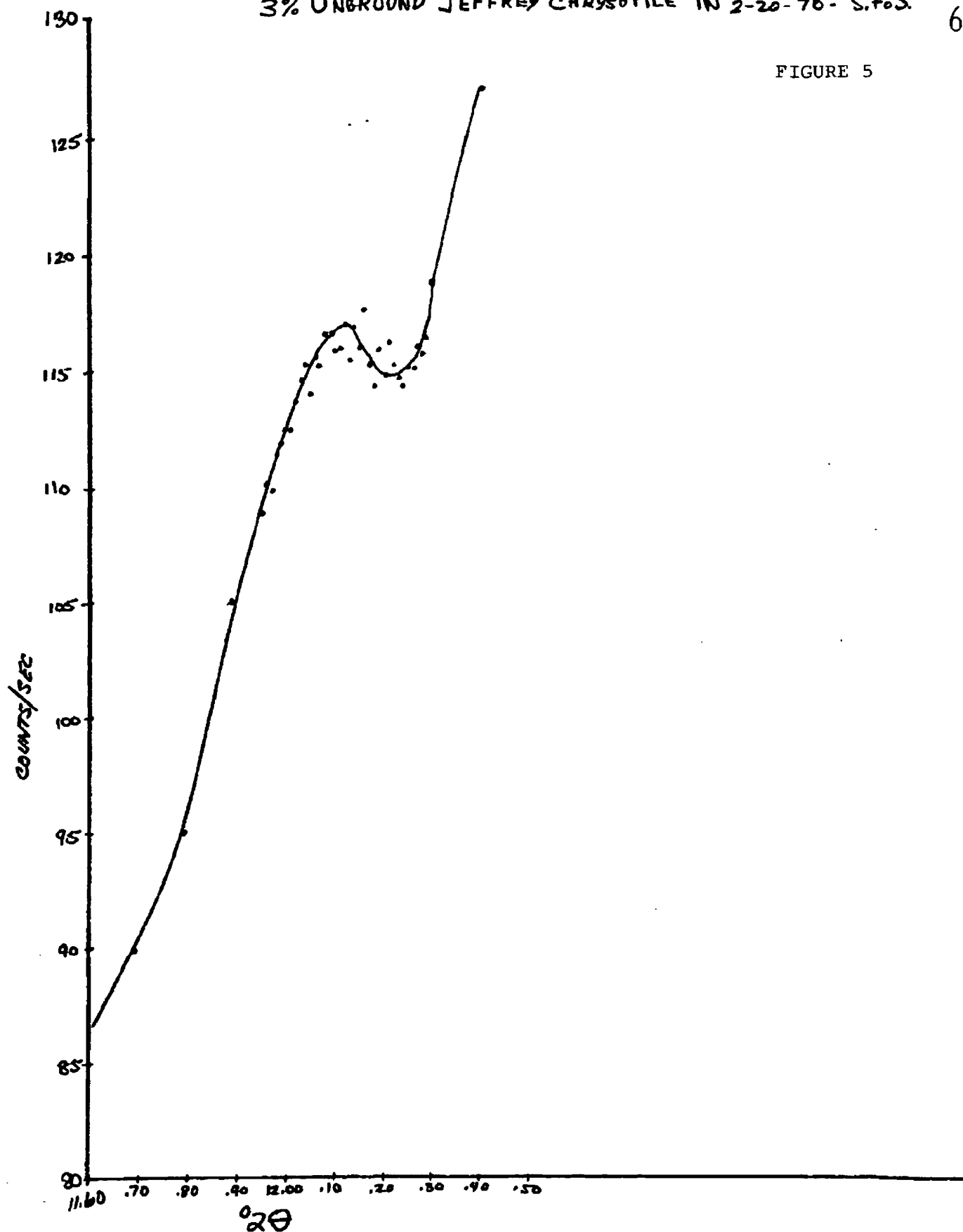


J&J-0130768

3% UNGROUND JEFFREY CHRYSOTILE IN 2-20-70 - S.toS.

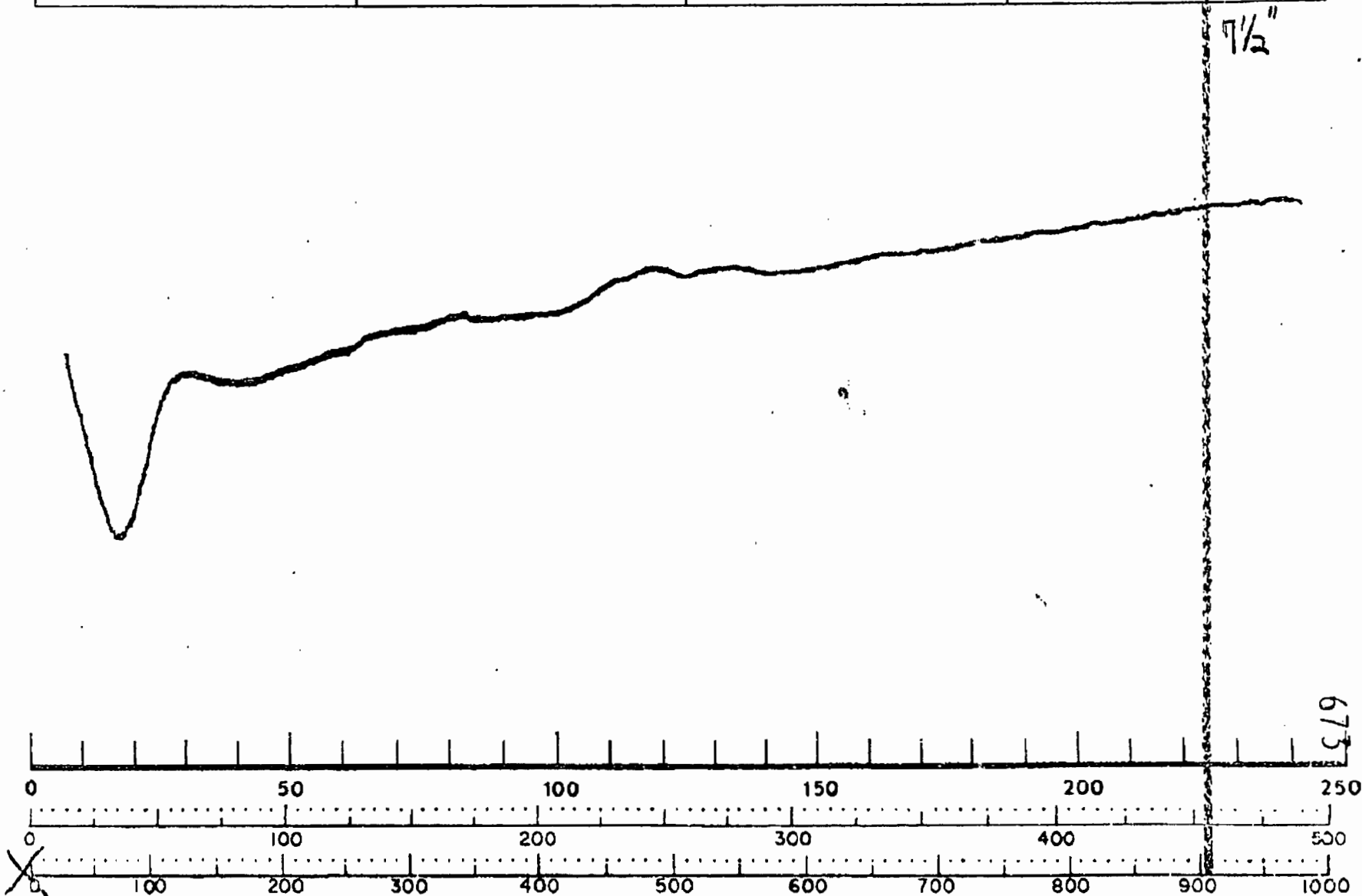
672

FIGURE 5



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SAMPLE <u>Baseline</u>	SIZE <u>PSH</u>	RANGE <u>40 μs</u>	RUN NO. _____
ORIGIN:	REF. <u>Alumina</u>	GAS 1. <u>He</u> 2. _____	DATE <u>9/11/77</u>
	PROG. MODE <u>Heat</u>	PRESSURE _____ lbs./in ²	OPERATOR <u>DM</u>
	PROG. RATE <u>10°C/min</u>	VACUUM _____ mm	REMARKS _____

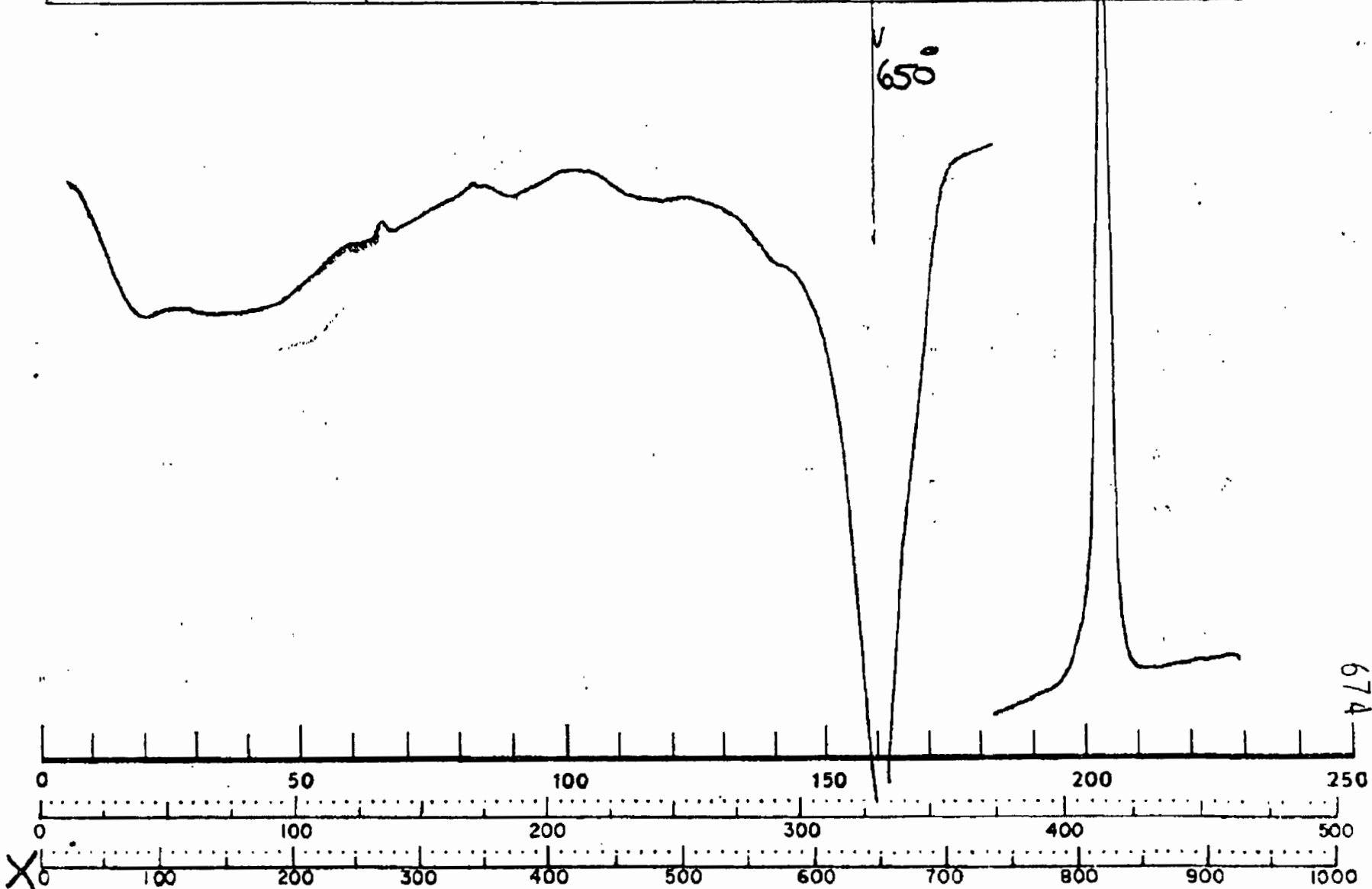


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Figure 6

SAMPL. CHRYSOPILE Diluted 75% in 325 mesh alumina (Oxal. sol.) ORIGIN:	SIZE <u>PSH</u>	RANGE <u>Kα 4</u>	RUN NO. _____
	REF. <u>Alumina</u>	GAS 1. <u>He</u> 2. _____	DATE <u>Apr 13, 1972</u>
	PROG. MODE <u>Heat</u>	PRESSURE _____ lbs./in ²	OPERATOR <u>M</u>
	PROG. RATE <u>10°C/min</u>	VACUUM _____ mm	REMARKS <u>82d 25%</u>

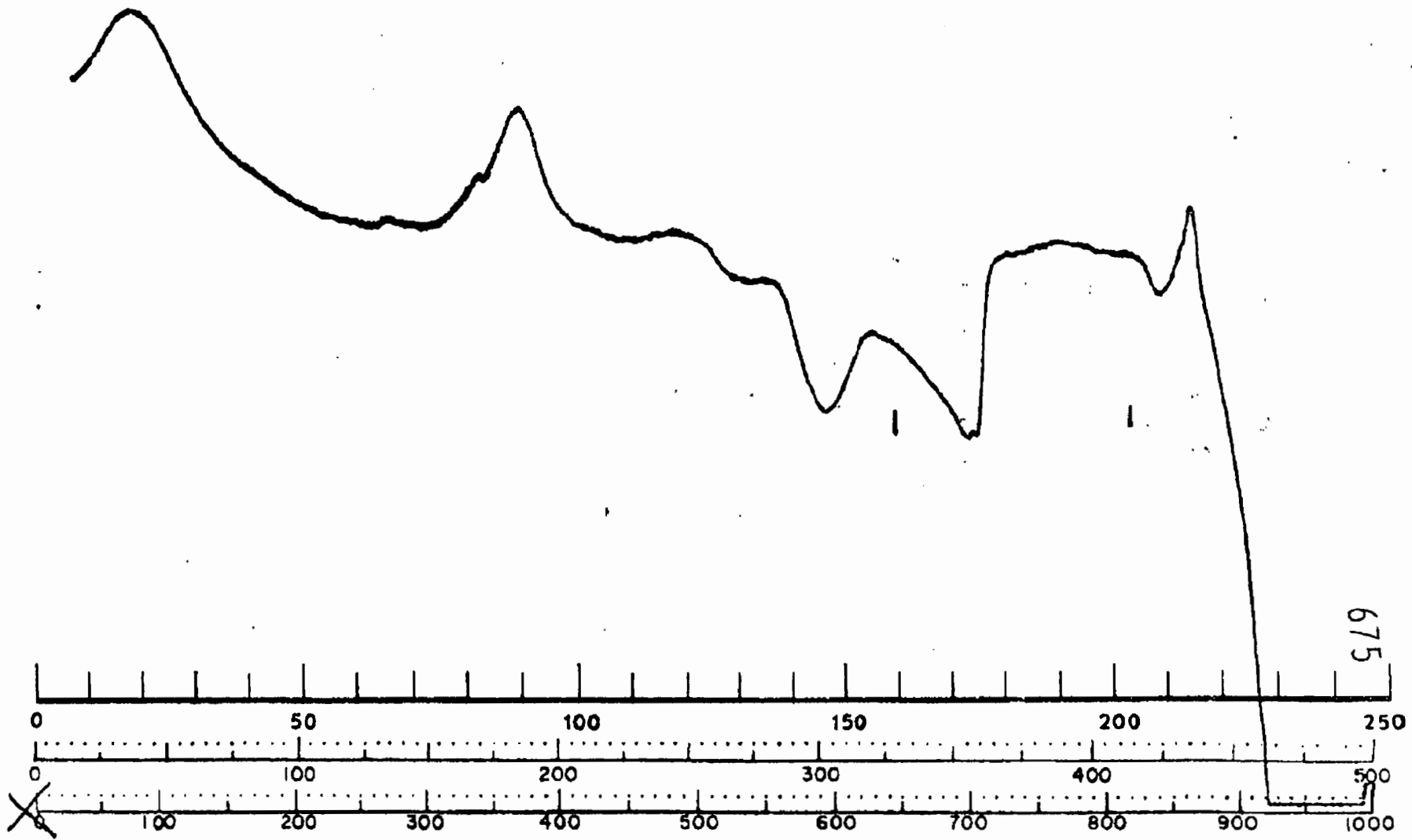


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JNJA265_000017791

Figure 7

SAMPLE <u>343 1615</u> <u>Lot # 82265</u> <u>Talc</u> ORIGIN:	SIZE <u>PSH</u>	RANGE <u>40 μm</u>	RUN NO. _____
	REF. <u>Alumina</u>	GAS 1. <u>He</u> 2. _____	DATE <u>9/12/72</u>
	PROG. MODE <u>Heat</u>	PRESSURE _____ lbs./in ²	OPERATOR _____
	PROG. RATE <u>10°C/min.</u>	VACUUM _____ mm	REMARKS _____

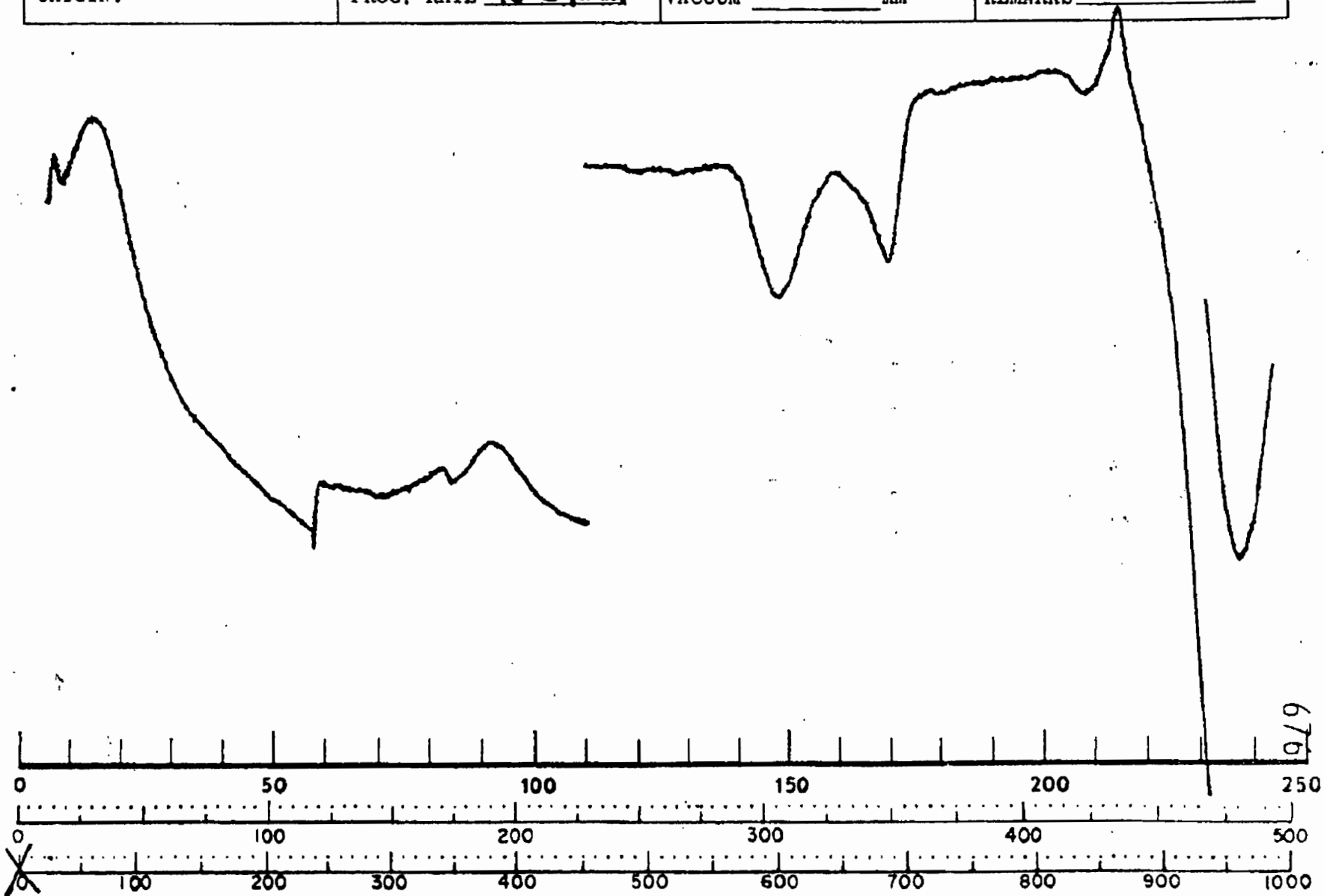


J8J-0130772

JN/AZ55_000017792

Fig 8

SAMPLE 006-OK 2-20-70 325 mesh Shown to Shover ORIGIN:	SIZE <u>PSH</u>	RANGE <u>402</u>	RUN NO. _____
	REF. <u>Aluminum</u>	GAS 1. <u>He</u> 2. _____	DATE <u>Aug 28, 1972</u>
	PROG. MODE <u>Heat</u>	PRESSURE _____ lbs./in ²	OPERATOR <u>M. M.</u>
	PROG. RATE <u>10°C/min</u>	VACUUM _____ mm	REMARKS _____

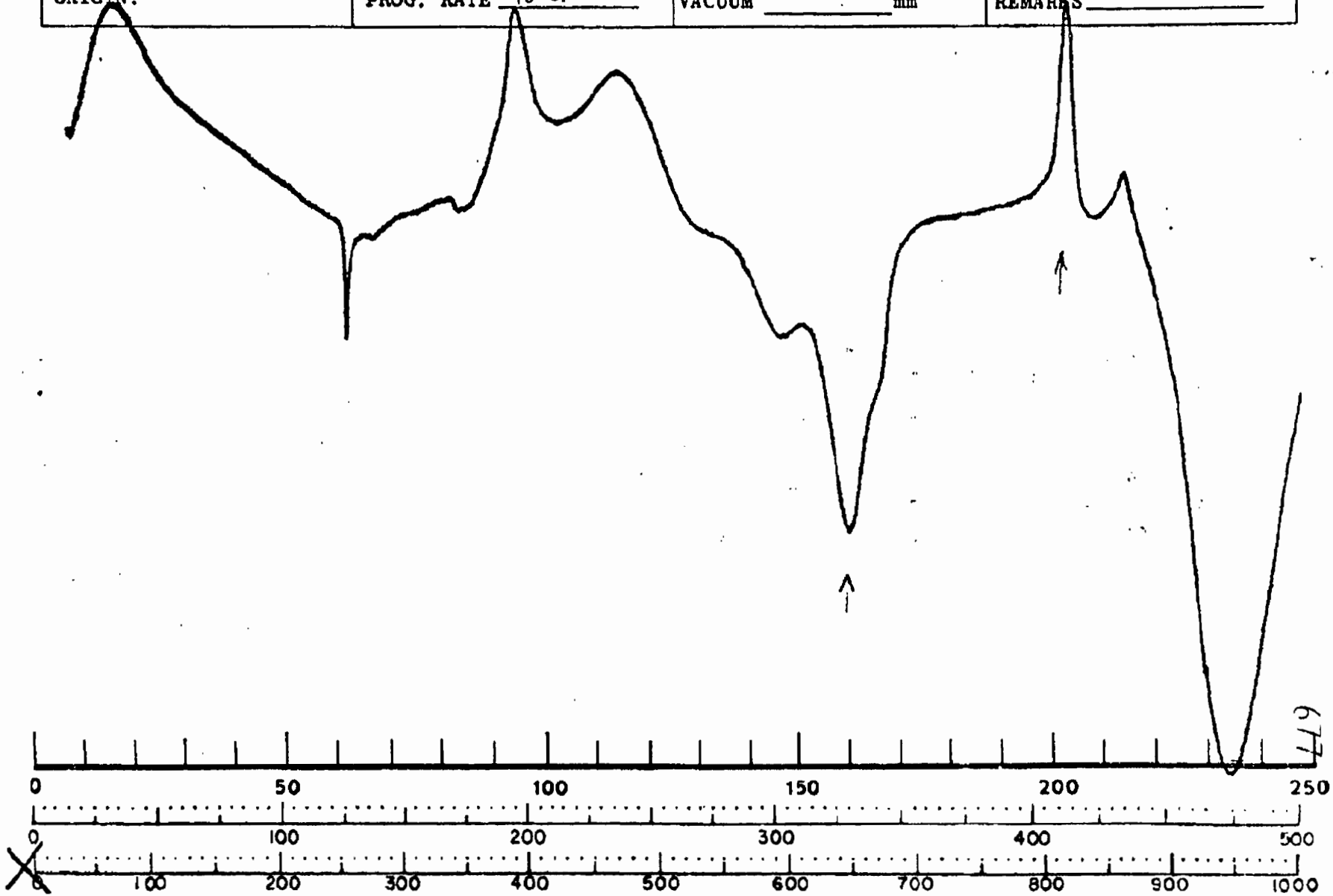


J8J-0130773

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Figure 1

SAMPLE. 5% UNDERGROUND JEFFREY CHRYSTLE in 2-26-70 sto S	SIZE <u>P. SH.</u> REF. <u>Alumina</u> PROG. MODE <u>Heat</u> PROG. RATE <u>10 °C/min</u>	RANGE <u>1024</u> GAS 1. <u>He</u> 2. <u> </u> PRESSURE <u> </u> lbs./in ² VACUUM <u> </u> mm	RUN NO. <u> </u> DATE <u>9-11-72</u> OPERATOR <u>M</u> REMARKS <u> </u>
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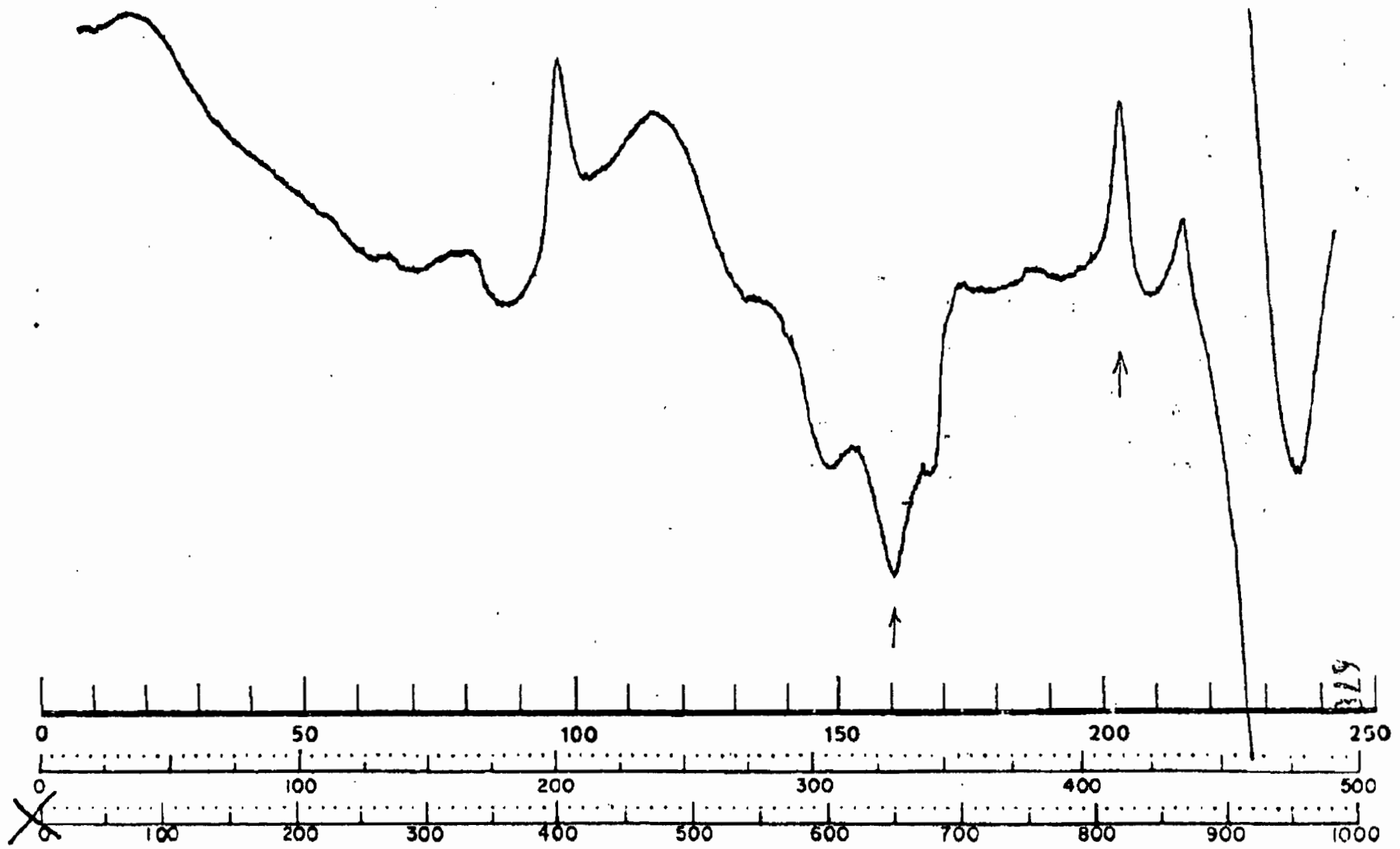


J&J-0130774

JNLAZ55_000017794

Figure 10

SAMPL 3% Jeffery chrysotile in 2-10-70 STAS ORIGIN:	SIZE <u>PSH.</u>	RANGE <u>40µr</u>	RUN NO. _____
	REF. <u>Alumina</u>	GAS 1. <u>He</u> 2. _____	DATE <u>9/14/72</u>
	PROG. MODE <u>Heat</u>	PRESSURE _____ lbs./in ²	OPERATOR _____ mm.
	PROG. RATE <u>10°C/min</u>	VACUUM _____ mm	REMARKS _____

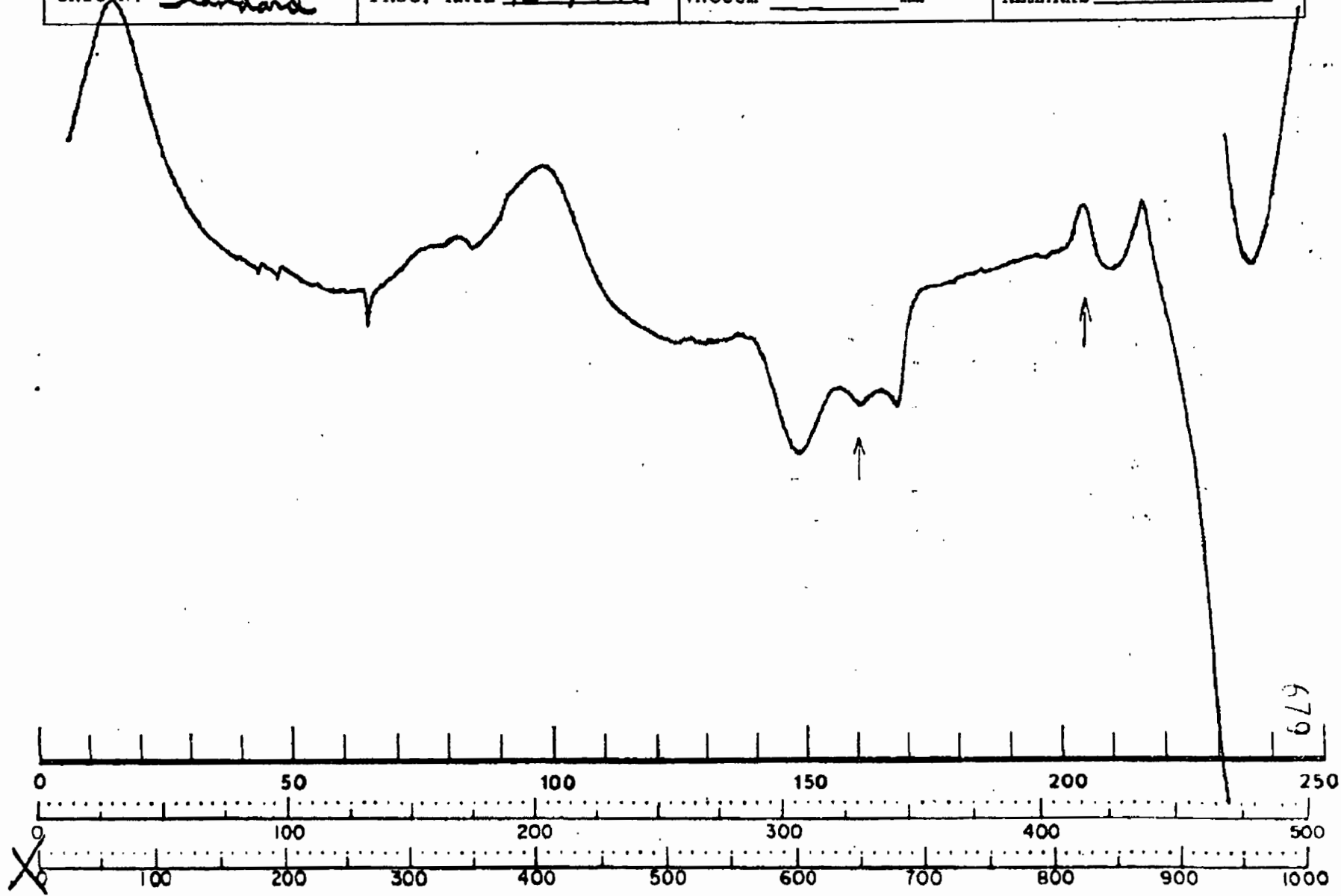


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JN/AZ55_000017795

Figure 11

SAMPLE <i>170 chrysotile in 2-20-70 shown to shown</i> ORIGIN: <i>Standard</i>	SIZE <i>PSH</i>	RANGE <i>40µm</i>	RUN NO. _____
	REF. <i>Alumina</i>	GAS 1. <i>He</i> 2. _____	DATE <i>8/30/72</i>
	PROG. MODE <i>Heat</i>	PRESSURE _____ lbs./in ²	OPERATOR _____
	PROG. RATE <i>10°C/min</i>	VACUUM _____ mm	REMARKS _____



J&J-0130776

JNJA265_000017796

21 mg

SAMPLE <i>Dr. Lewin's</i> <i>Shower to Shower</i>	SIZE <i>PSH</i>	RANGE <i>40µv</i>	RUN NO. _____
ORIGIN:	REF. <i>Alumina</i>	GAS 1. <i>He</i> 2. _____	DATE <i>9/13/72</i>
	PROG. MODE <i>Heat</i>	PRESSURE _____ lbs./in ²	OPERATOR _____
	PROG. RATE <i>10°C/min.</i>	VACUUM _____ mm	REMARKS _____

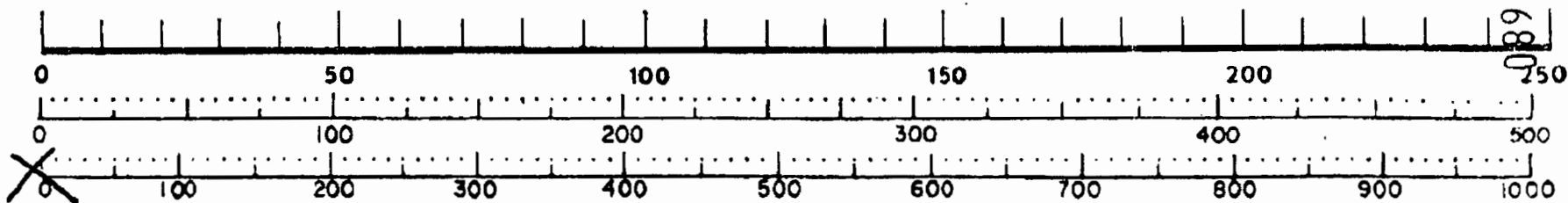
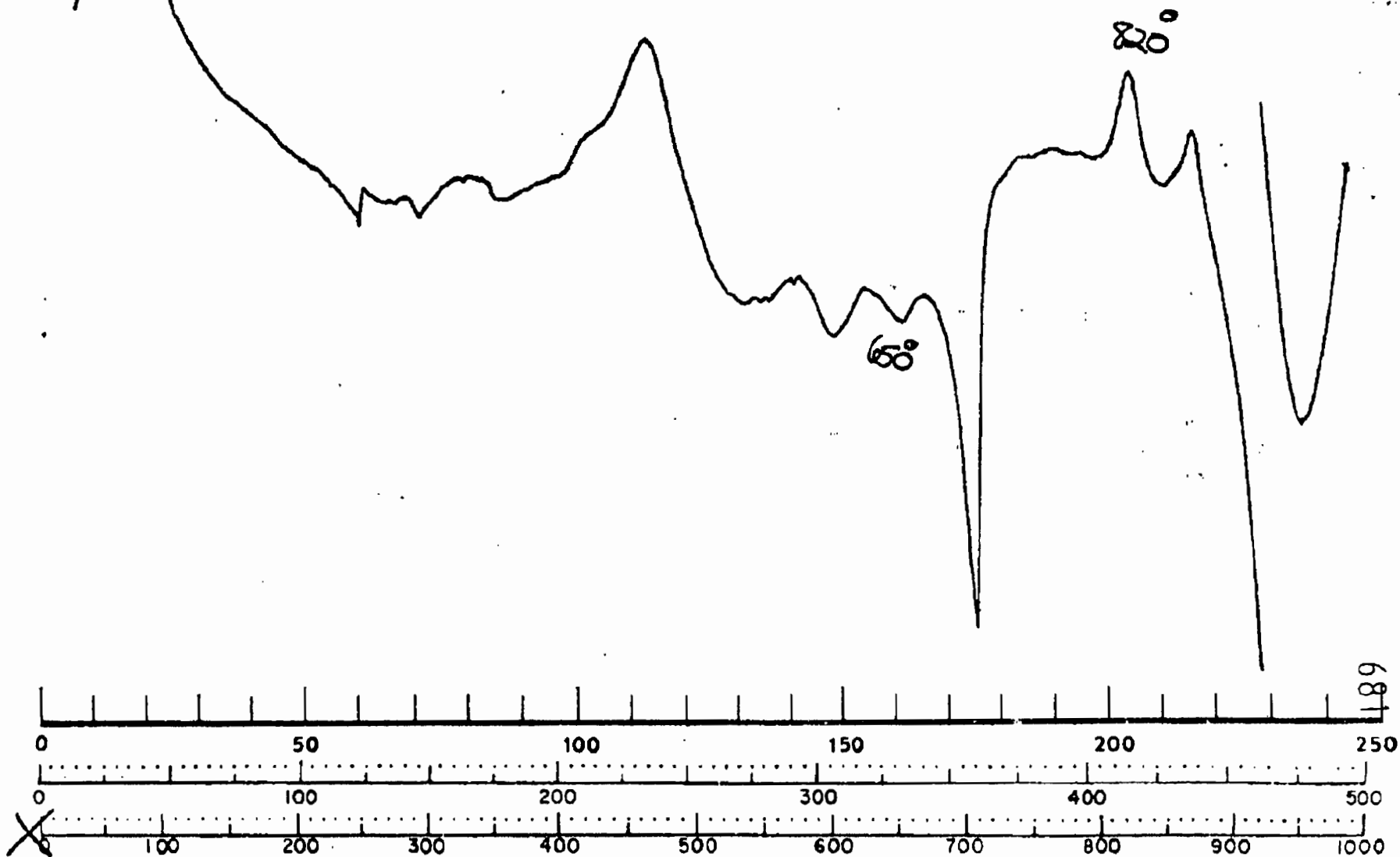


Figure 13

SAMPLE Pb chloride in Dr. Lewin's sample Shower to Shower	SIZE <u>PSH</u>	RANGE <u>40μr</u>	RUN NO. _____
ORIGIN	REF. <u>Alumina</u>	GAS 1. <u>He</u> 2. _____	DATE <u>8/31/72</u>
	PROG. MODE <u>Heat</u>	PRESSURE _____ lbs./in ²	OPERATOR <u>M.M.</u>
	PROG. RATE <u>10°C/min.</u>	VACUUM _____ mm	REMARKS _____



J8J-0130778

JN/AZ55_000017798